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Project No. 6031.2

1. Contractor's Name and Address:

Springborn Laboratories, Inc.
Department of Analytical Chemistry
Ten Springborn Center
Enfield, CT 06082

2. Title of Report:

Development of Acceptance Criteria for Batches of Silane
Primer for External Tank Thermal Protection System
Bonding Applications

March 2, 1985 - May 24, 1985

3. Date of Publication:

April 10, 1985

4. Type of Report and Contract Number:

11th Progress: NAS8-35818

5. Authors:

F. Mikes, C. Mowrey, E. Reis

6. Prepared For:

C. Marshall Space Flight Center, AL 35812

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AS24D - 3x
AT01 - 1x
EM13B-18/Belvins - 1x
EH33/Morris - 10x
NASA Scientific & Technical Info. Facility - 1x + Repro.



(NASA-CR-175850) DEVELOPMENT OF ACCEPTANCE
CRITERIA FOR BATCHES OF SILANE PRIMER FOR
EXTERNAL TANK THERMAL PROTECTION SYSTEM
BONDING APPLICATIONS Progress Report, 2
Mar. - 24 May 1985 (Springborn Labs., Inc., G3/27

N85-26995

Unclass
21390

TABLE I*

DC 1200 Primer Lots Used in Project Program

<u>Springborn Labs ID #</u>	<u>DC Primer Lot #</u>	<u>Date Received at Springborn</u>	<u>Additional Description and Further Sample Labeling</u>
A**	QL033703	5/11/83	red opaque
B**	QL071621	11/29/83	red, leaked
C**	QL093752	11/29/83	clear, sealed
1***	EN057367	3/30/84	clear
2***	QL033705	3/30/84	red
3***	063711	3/30/84	red, S/L Exp 6/84, Temp 50/90F RS3.900324, 7/83 MMSK343A025 83G382
4***	093713	3/30/84	red, S/L Exp. 09/84, Temp 50/90F, RS3.794481 09/83 MMSK343A025 83G530 (hold for J Mills)
5***	093733	3/30/84	red, S/L Exp 09/84, Temp 50/90F, RS3.794481, 09/83 MMSK34A025 83G529
6***	071620	3/30/84	red, S/L Exp 7/82, Temp 50 to 90F, RS1:705481, 08/81 MMSK343A025 81G464
7 ⁺	QL103753	1/10/85	red

*A sample identification table is included as the first page in each report.

**Three (3) DC 1200 Primer Lots acquired by Springborn Laboratories for initial tests (see monthly reports 1, 2 and 3).

***Six (6) DC 1200 Primer Lots received from NASA, Management Division Bldg. 4471, on March 30, 1984.

⁺DC 1200 Primer Lot received from NASA, Bldg. 4612, Mr. Don Morris, Project Monitor.

1. SHEAR TESTS - VALIDATION STUDY

From a series of 10 DC 1200 primers two silane primers (#3 and #7) were selected for validation studies. Recent FTIR analyses (5/16/85, Figure 1) indicated that sample #3 contains the highest OH-absorption area (74) and sample #7 the smallest OH value (12)*.

Lap sheer tests were performed on 17 metal strips coated with primers #3 and 13 metal strips coated with primer #7, respectively. All lap shear values obtained are reported with Mean, Standard Deviation and Relative Standard Deviation calculation in graphical and tabular form in Figure 2. The recordings obtained on the Instron instrument are included in Figure 3 (panels L, M, N) and Figure 4 (panels O and P). A detailed description of the shear tests is given in Figure 5a-9b.

2. LAP SHEAR TESTS OF SEVEN NASA SILANE PRIMERS vs. INFRARED TESTS FOR OH - ABSORPTION.

2.1 Silane Aging vs OH-Absorption

A series of DC 1200 primers were analyzed four times during 1984-85 for their OH-Absorption areas and are enclosed in graphic form for each silane in Figures 10 to 19.

Silane samples deteriorated during the year (although covered with a dry nitrogen layer before closing the metal containers, some moisture and air can enter during opening). An OH-absorption value of ≥ 80 ** indicates almost total hydrolysis of silane batch (i.e. sample slightly opaque if red dye added). The new silane batches OH-absorption values were found to be between 0 and 3. The average values for OH-areas are included in Figures 10 to 19.

The most recent FTIR spectra (May'85) for all ten (10) silane primers are included as Figures 20-29.

* In this report all four OH-absorption band values, given separately in previous reports, were averaged and only one absorption value is given. Areas were calculated using the Trapezoid Rule, where the area A_{OH} is

$$A_{OH} = \int_{a=3470 \text{ cm}^{-1}}^{b=3360 \text{ cm}^{-1}} f(x) dx = \left(\frac{Y_0}{2} + Y_1 + Y_2 + Y_3 + \frac{Y_4}{2} \right) \Delta X$$

$$\text{AND } \Delta X = \frac{b-a}{n} = \frac{110 \text{ cm}^{-1}}{4} = 27.5 \text{ cm}^{-1}$$

** Area calculated using Trapezoid Rule.

2.2 Correlation of OH-Absorption of Silane Primers with Lap Shear Tests of Coated Panels

FTIR area values of OH-absorptions recently obtained (5/16/85) were plotted against lap shear values (mean values) of the DC 1200 primer used for adhesive bonding between two aluminum panels (coated with deSoto primer). (The bonding procedure was described in the 10th report, pg. 3).

The graphic presentation of FTIR and shear test values is given in Figure 30. Individual values for each lap shear test are given in tables for test series R to Z in Figures 31a to 39b. The statistical values for panel tests bonded with DC 1200 silane primers #1 to 7 are summarized in the table below.

Statistical Values for Lap Shear Tests of Seven Silane Primer Lots

Silane Lot No.	n (Total of Lap Shear Tests)	M (Mean)	STD (Standard Deviation)	RSTD (Relative STD)	Fig. No.
1	10	136	16	12	5-9, 31, 33, 34
2	11	82	26	32	31, 32, 35
3	21	60	22	35	5-9, 39
4	10	76	16	21	36, 37, 38
5	11	110	16	15	36, 37, 38
6	10	162	32	20	32, 33, 34
7	24	217	60	28	5-9, 35, 39

The lap shear test records for series of panels R, S, T, U, V, W, X, Y, and Z are included as Figures 40-48.

3. PLAN FOR FUTURE WORK

3.1 GC Analysis of Silane Primers (12th Progress Report)

Complete GC analyses of headspace polar volatiles evaluated for their alcoholic faction. This section is before completion and will be issued as the 12th Progress Report.

3.2 Final Report

A Final Report will be issued in June summarizing results obtained during the contract work.

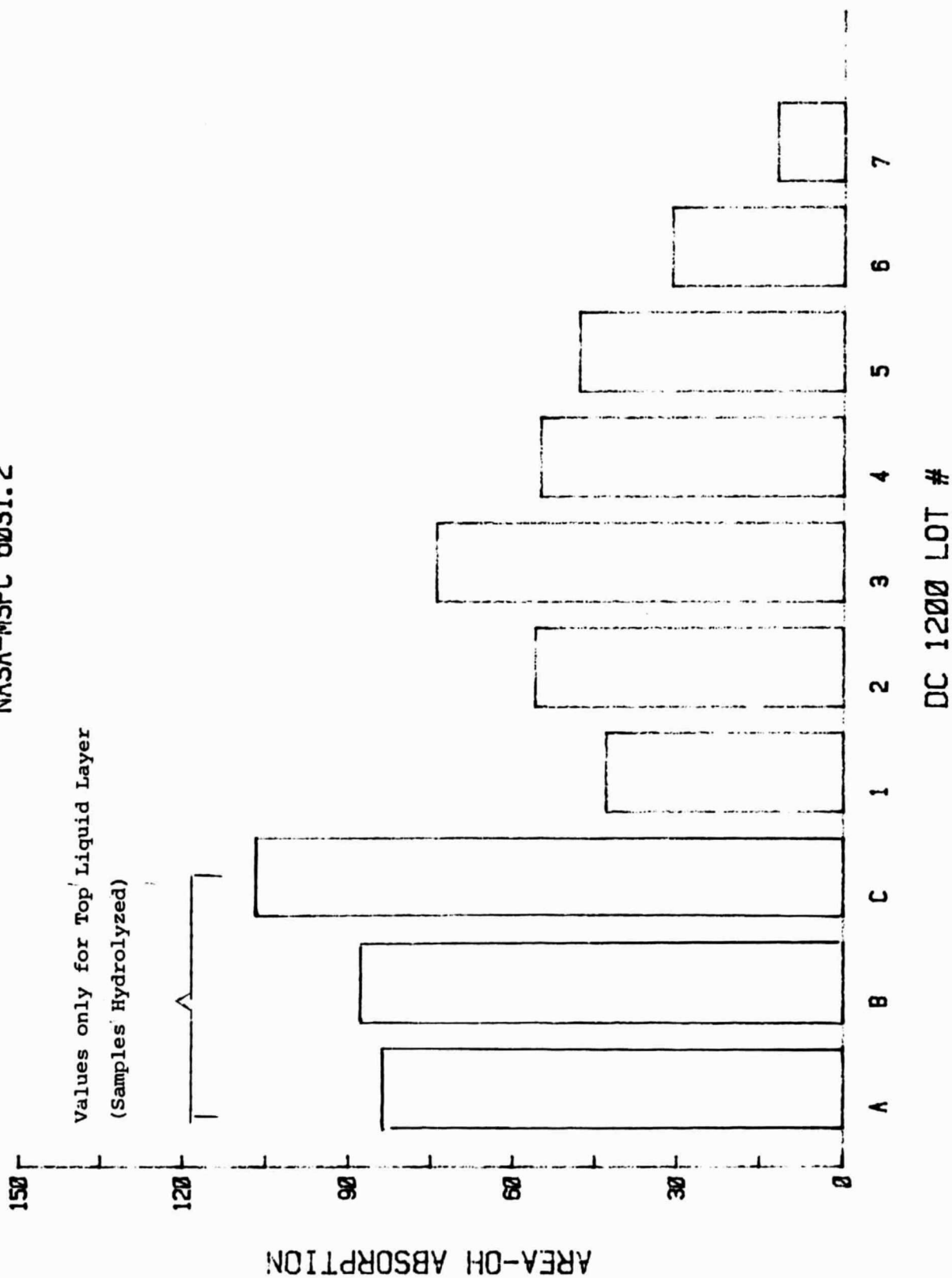
4. FINANCIAL STATUS

The total cumulative expenditure incurred as of report dated 1/3/85 to 92% of the total budget.

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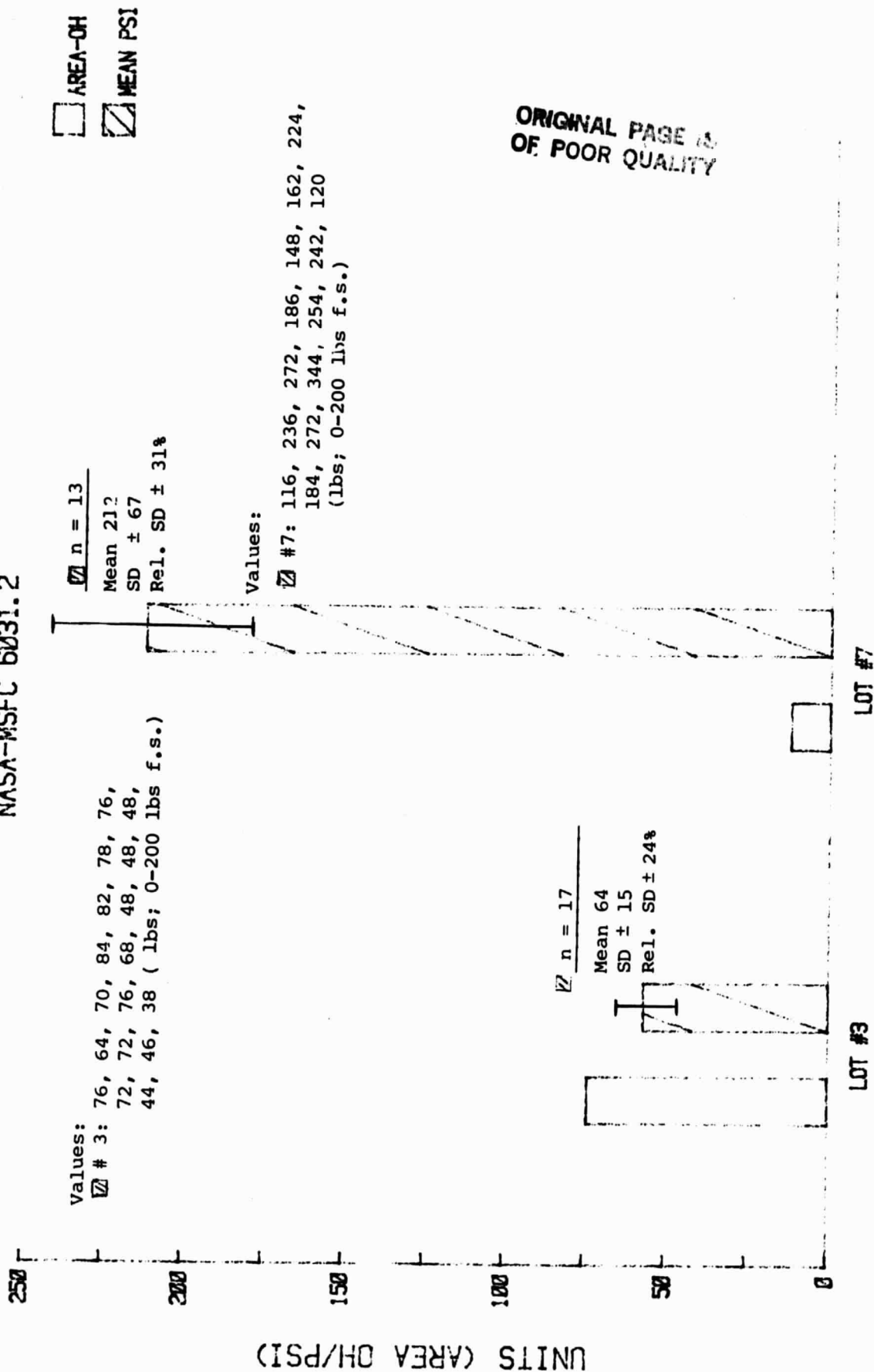
DC 1200 LOTS- 5-16-85

NASA-MSFC 6031.2



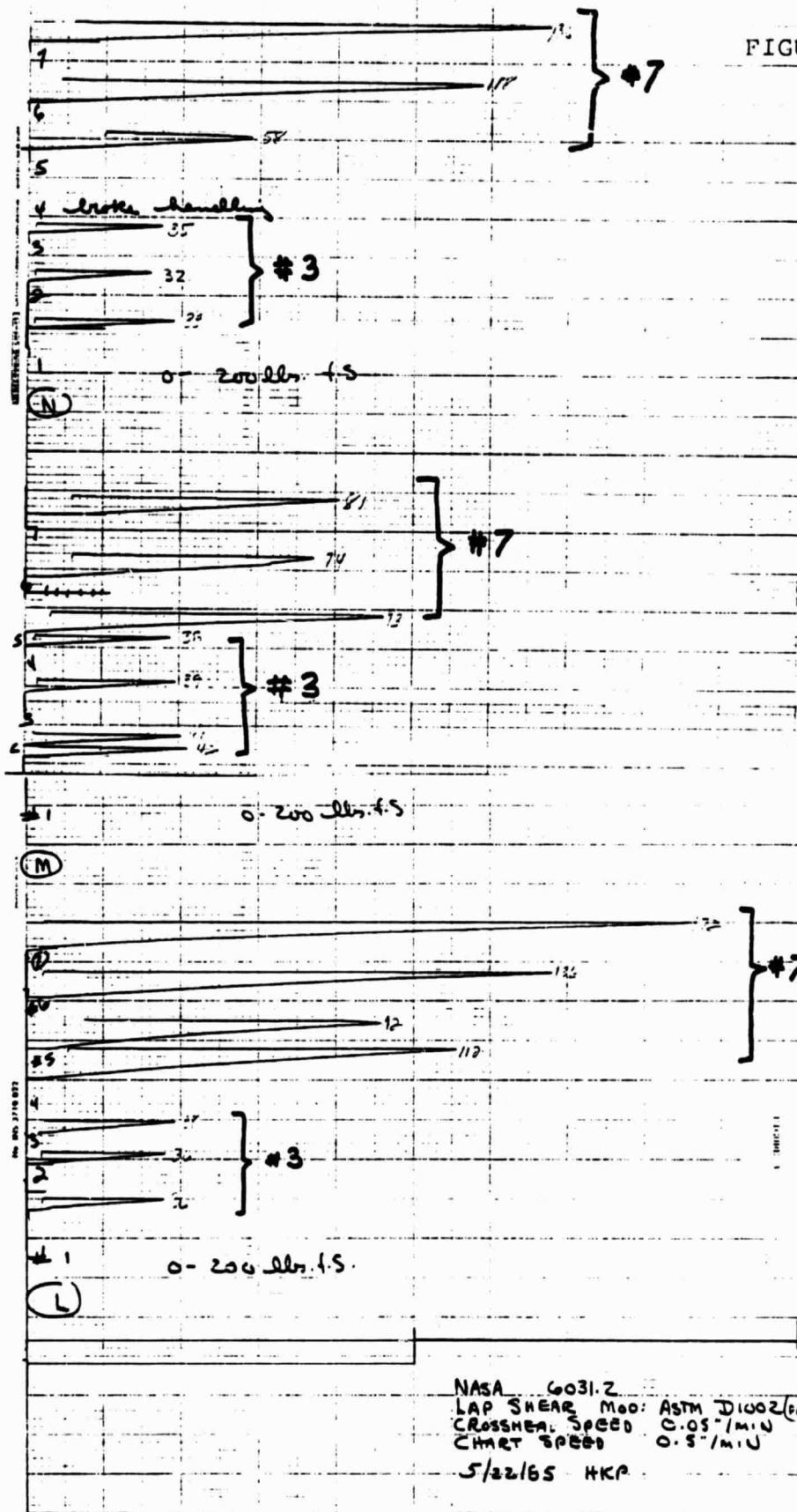
IR SPECTRUM VS SHEER TEST

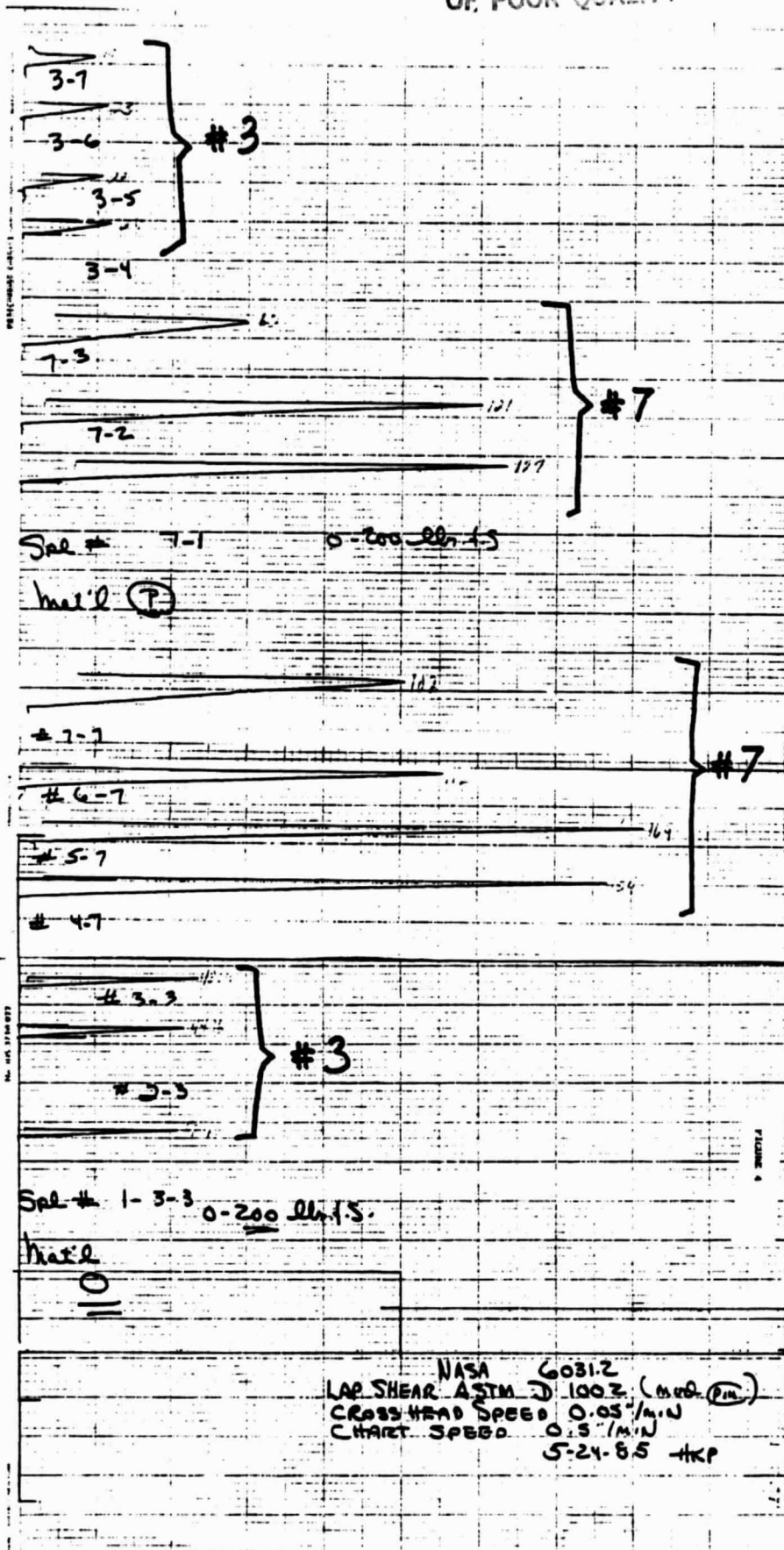
NASA-MSFC 6031.2



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FIGURE 3





SERIES PANEL L

NASA-MSFC
6031.2

0	0	0	0	0	0
3	3	7	7	7	7
1	3	9	5	6	7

X

BANYA CHAIYIN LANGRATH-4 S PC

LAP SHEAR TESTS

COMMENTS:

NASA-MSFC
6031.2

M

SERIES PANEL

0	0	0	0	0	0	0
3	3	3	7	7	7	7
1	2	3	4	5	6	7

✓

FIGURE 7a

SPRINGBOIN LABORATORIES, INC.

LAP SHEAR TESTS

COMMENTS:

NASA-MSFC
6031.2

N

SERIES PANEL

0	0	0	0	0	0	0
1	2	3	4	5	6	7
3	3	3	3	7	7	7
1	2	3	4	5	6	7

X

5-24-85

LAP SHEAR TESTS

[illegible]

COMMENTS:

SERIES PANEL 0

NASA-MSFC
6031.2

0	0	0	0	0	0	0
3	3	3	7	7	7	7
0						
1	2	3	4	5	6	7

LAP SHEAR TESTS

COMMENTS:

NASA-MSFC
6031.2

SERIES PANEL P

0	0	0	0	0	0
7	7	7	3	3	3
1	2	3	4	5	6
					7

AGING OF DC 1200 LOT # 1

NASA-MSFC 6031.2

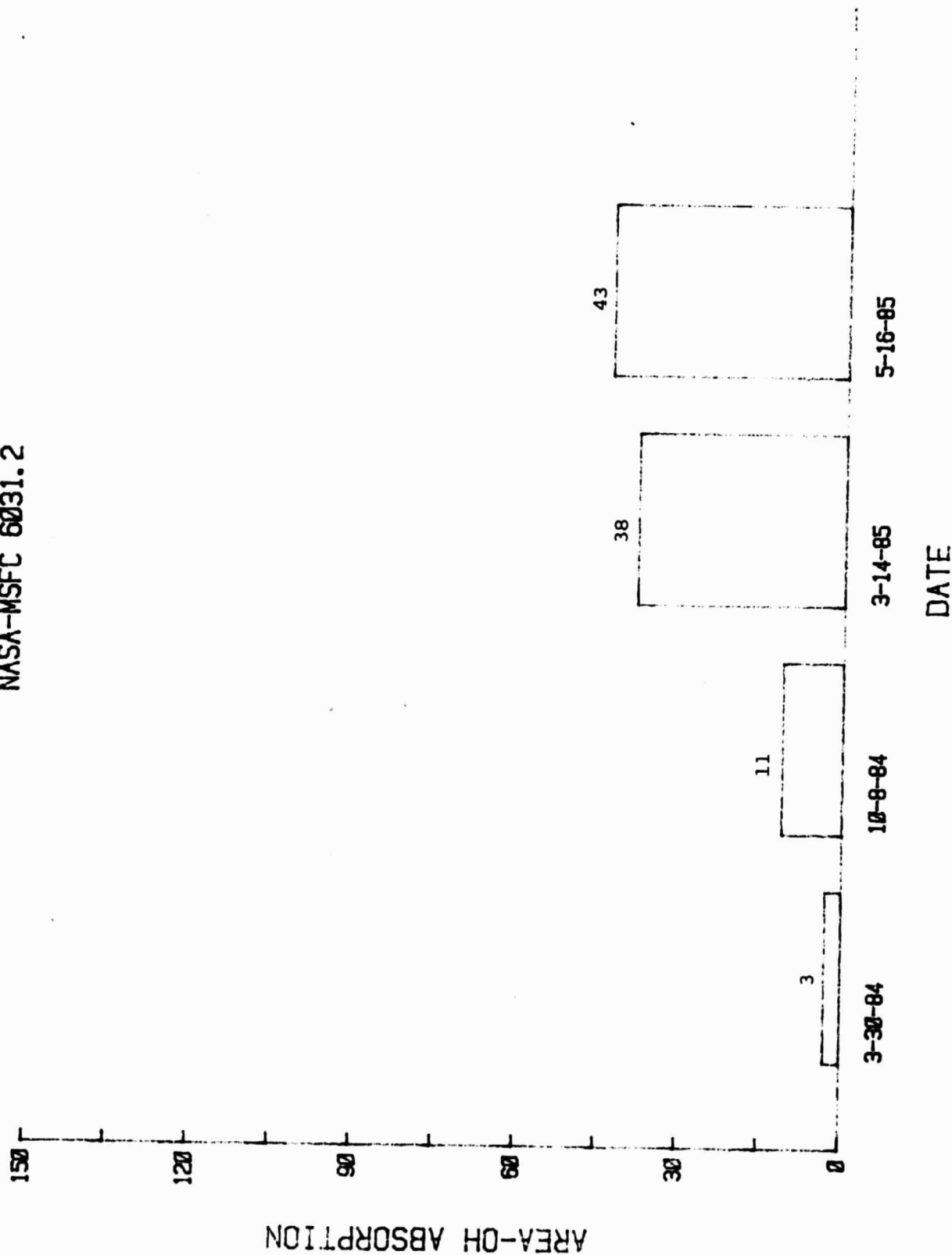


FIGURE 10

AGING OF DC 1200 LOT # 2

NASA-MSFC 6031.2

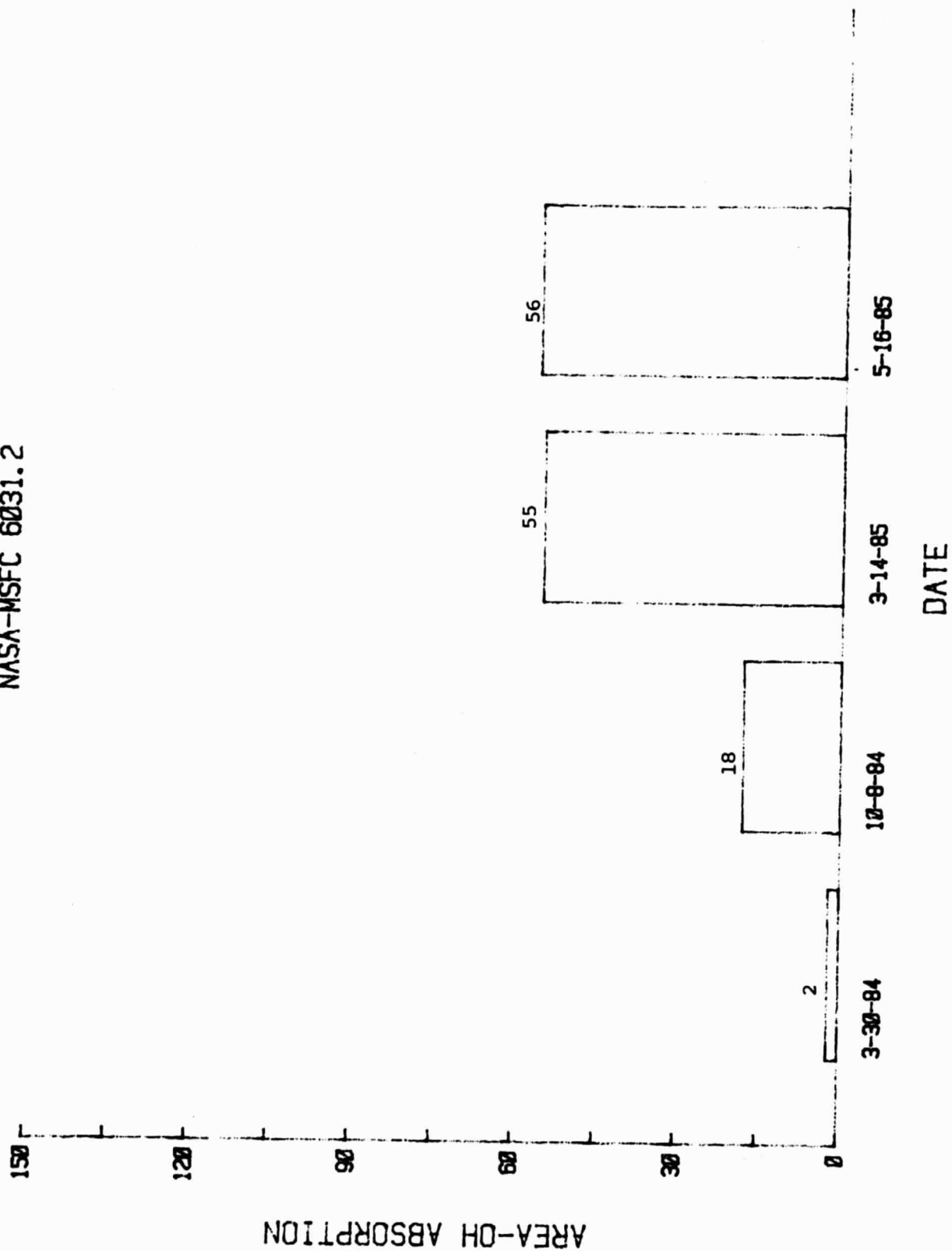
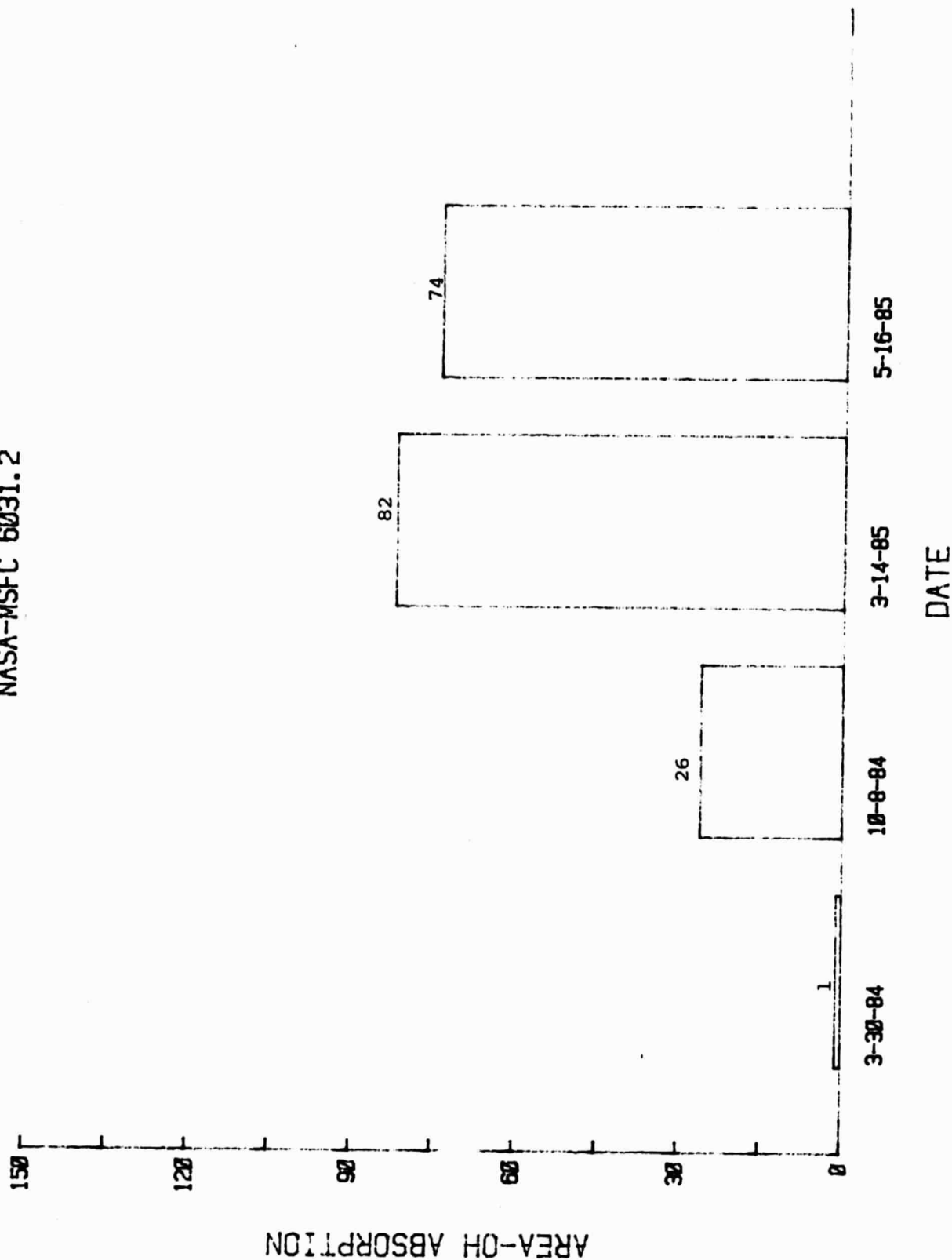


FIGURE 11

AGING OF DC 1200 LOT # 3

NASA-MSFC 6031.2



AGING OF DC 1200 LOT # 4

NASA-MSFC 6031.2

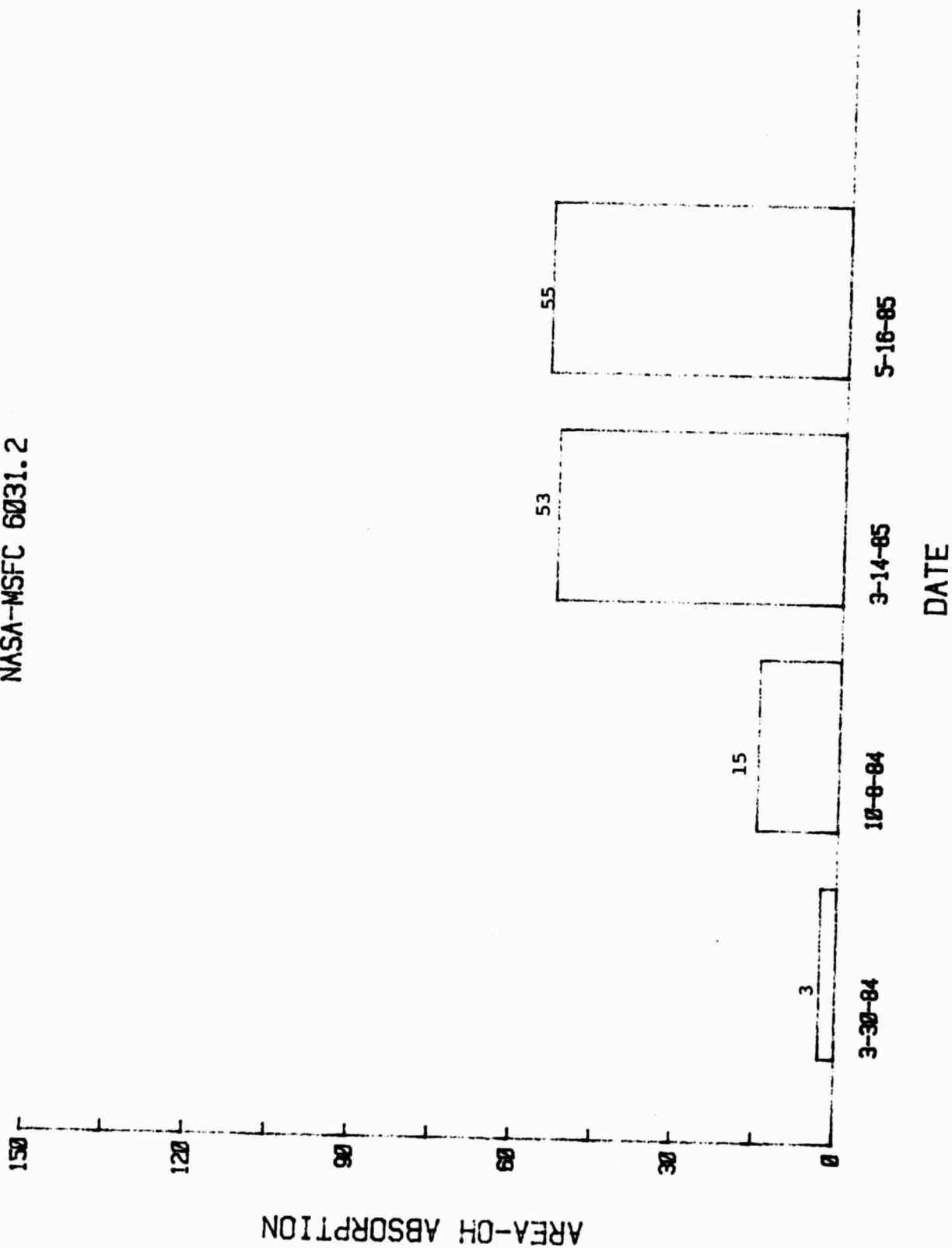


FIGURE 13

AGING OF DC 1200 LOT # 5

NASA-MSFC 6031.2

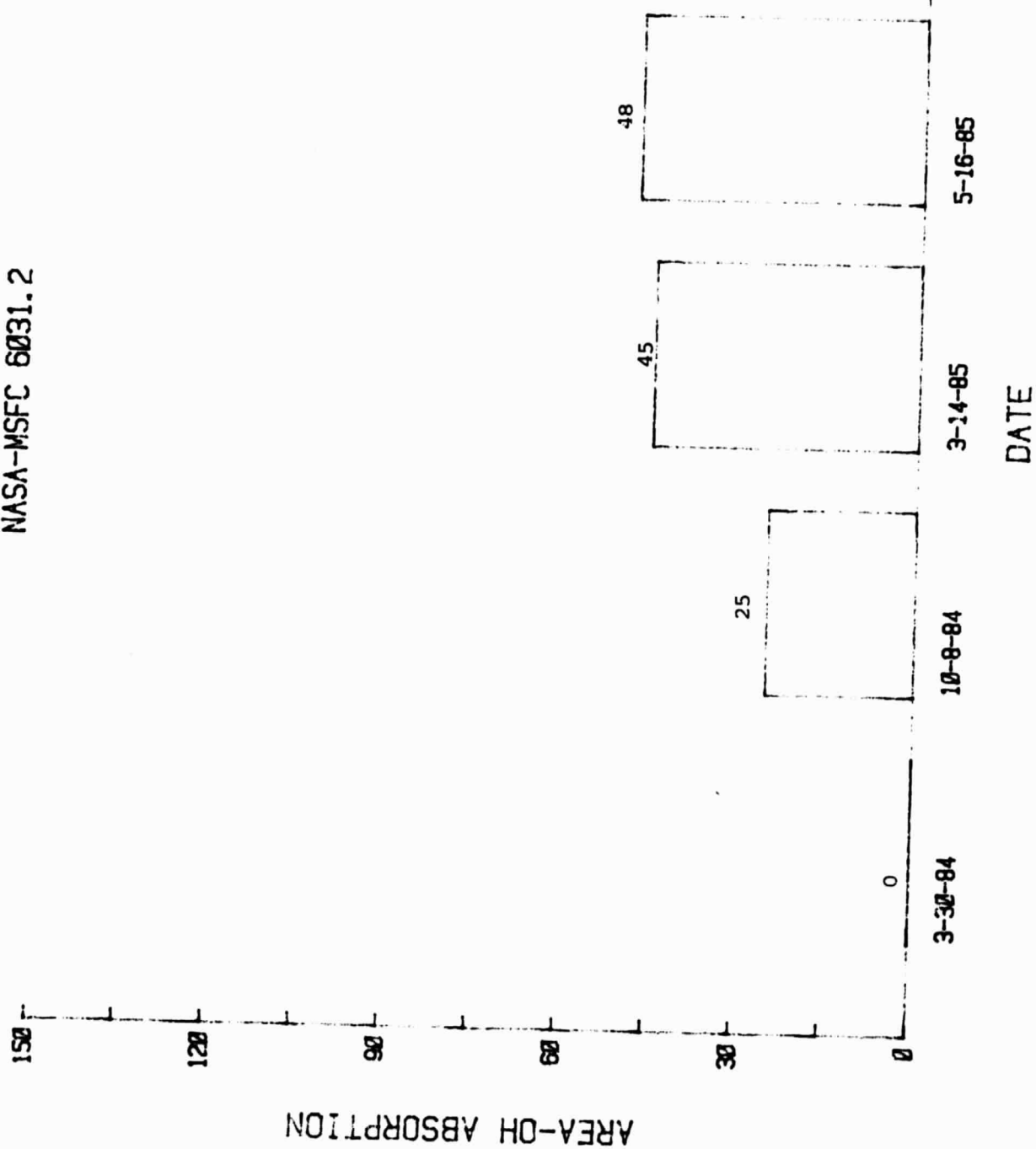
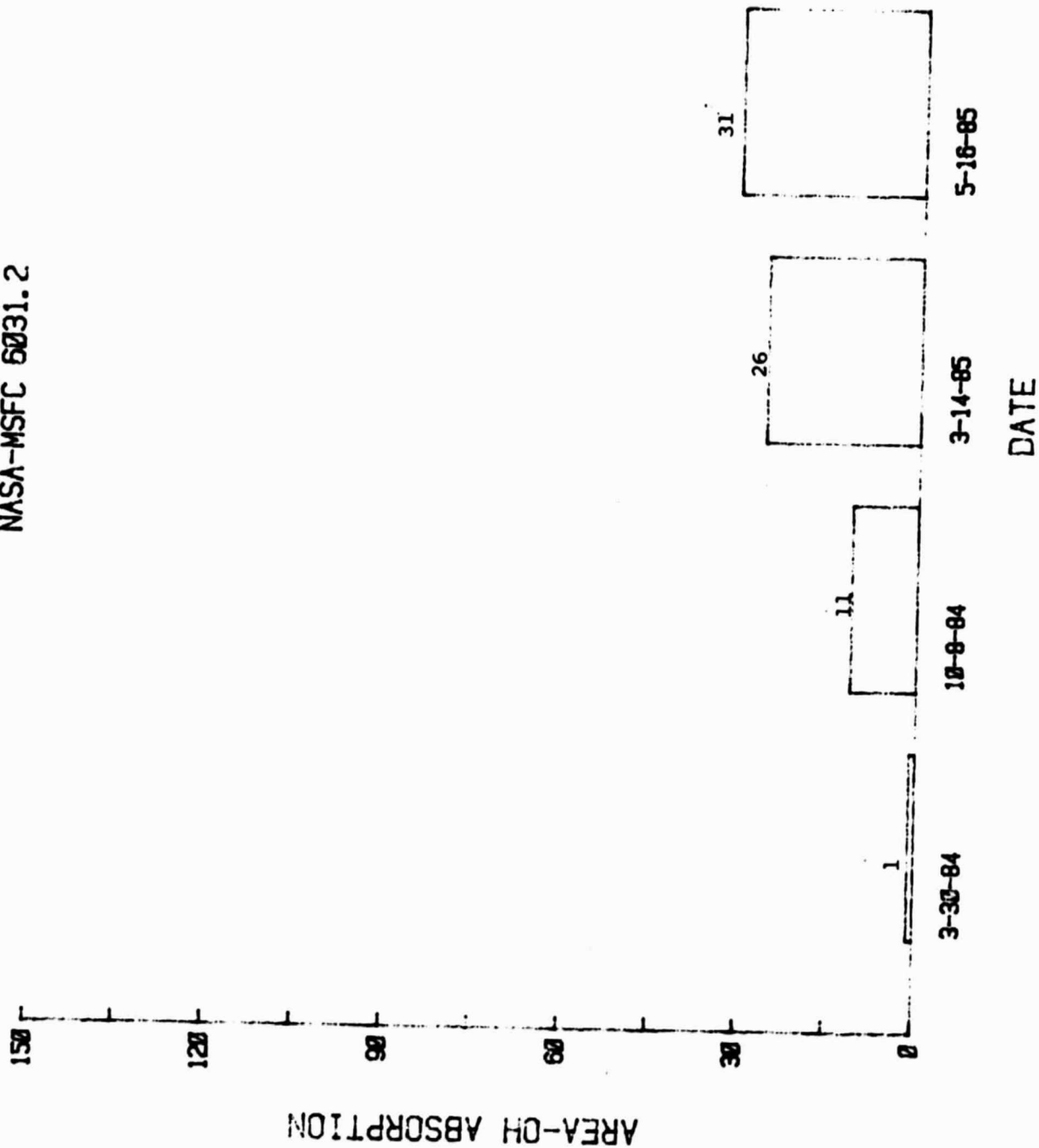


FIGURE 14

AGING OF DC 1200 LOT #6

NASA-MSFC 6031.2

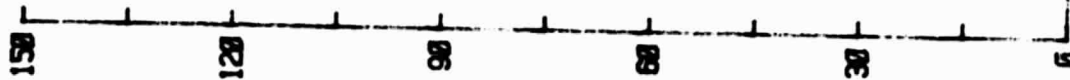


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FIGURE 15

AGING OF DC 1200 LOT # 7

NASA-MSFC 6031.2



AREA-OH ABSORPTION

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1/6/85
from NASA

12

h

3-30-84

10-9-84

3-14-85

5-16-85

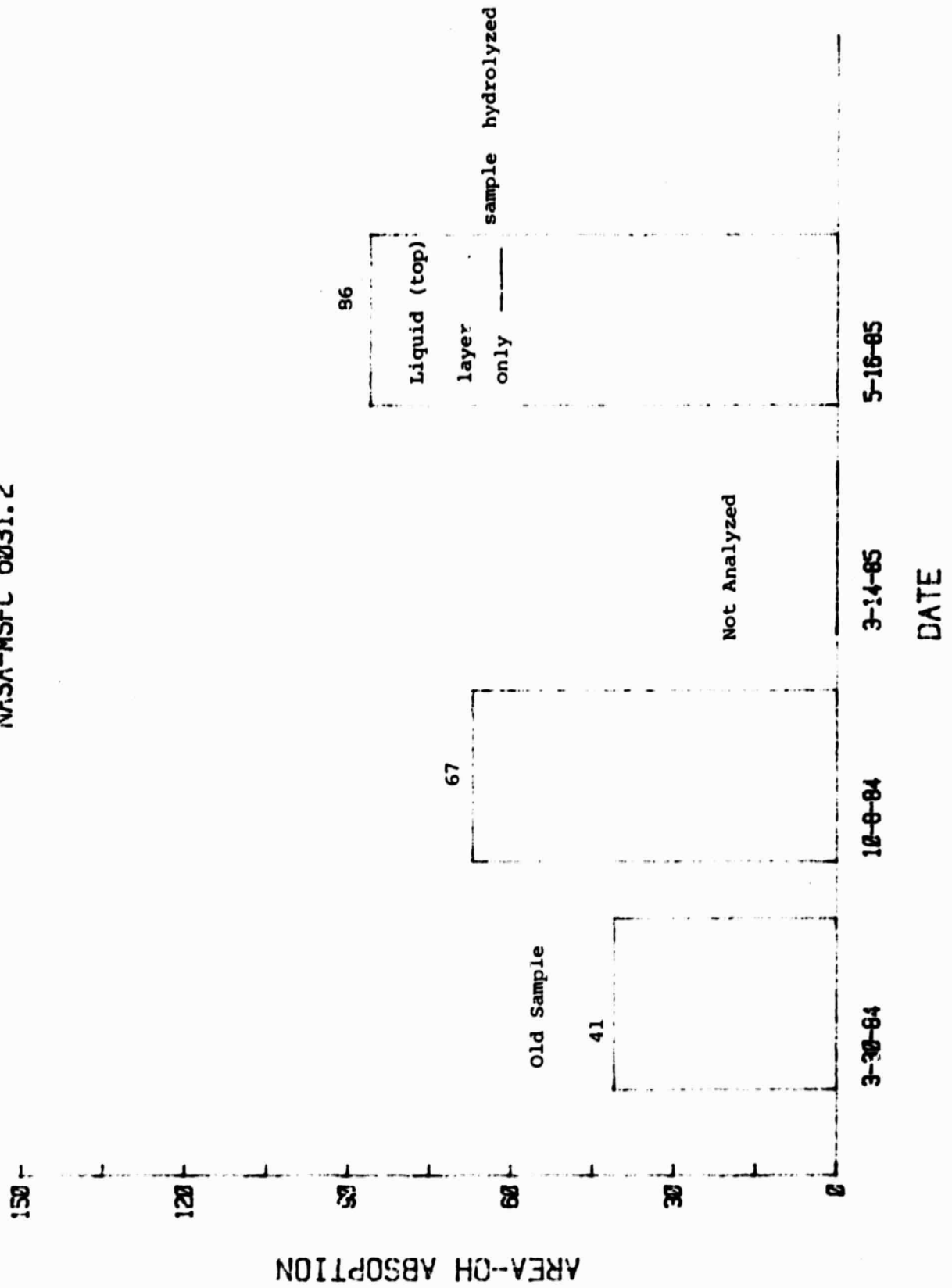
DATE

FIGURE 16

AGING OF DC 1200 LOT # A

NASA-MSFC 6031.2

FIGURE 17



AGING OF DC 1200 LOT # B

NASA-MSFC 6031.2



AGING OF DC 1200 LOT # C

NASA-MSFC 6031.2

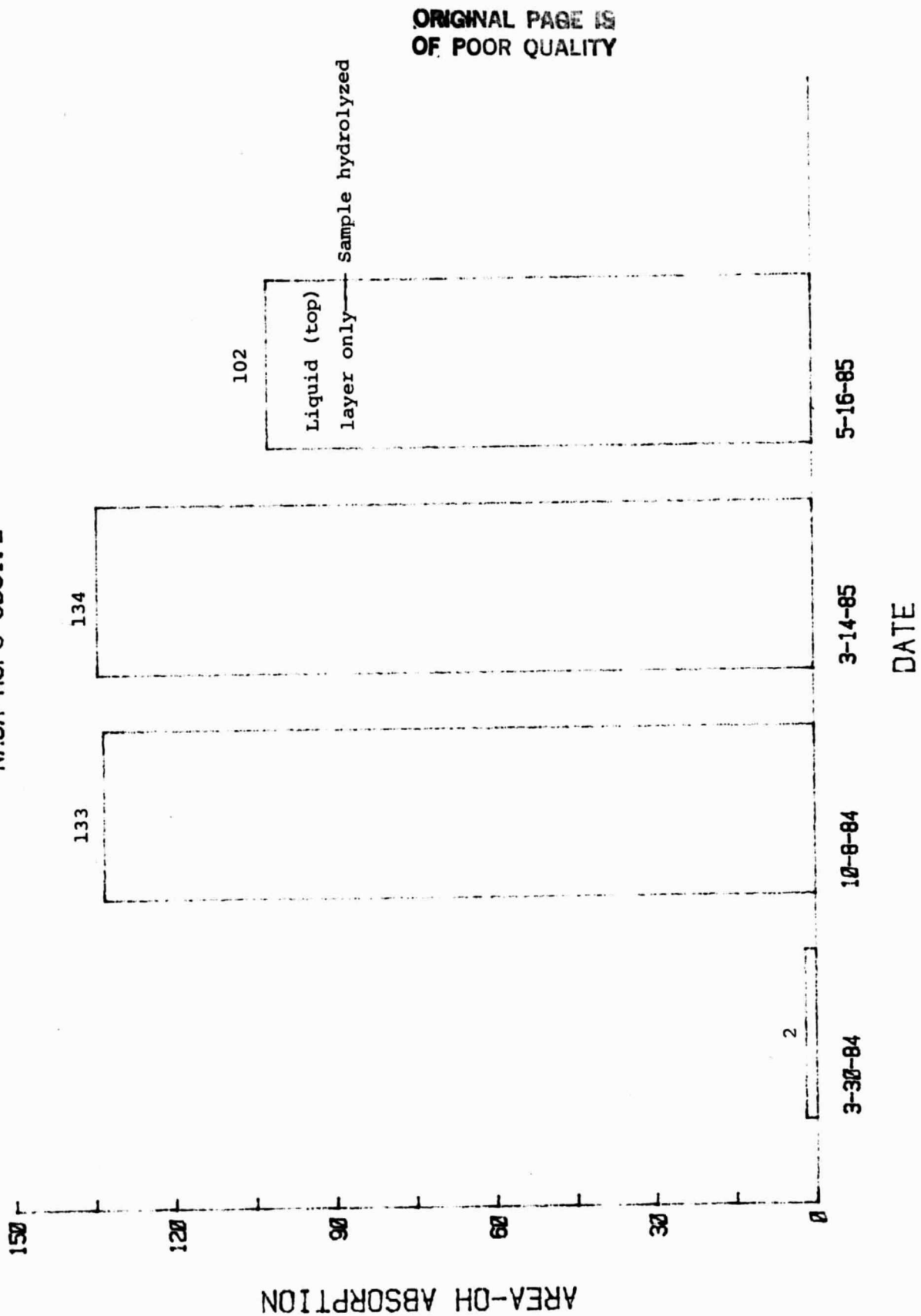
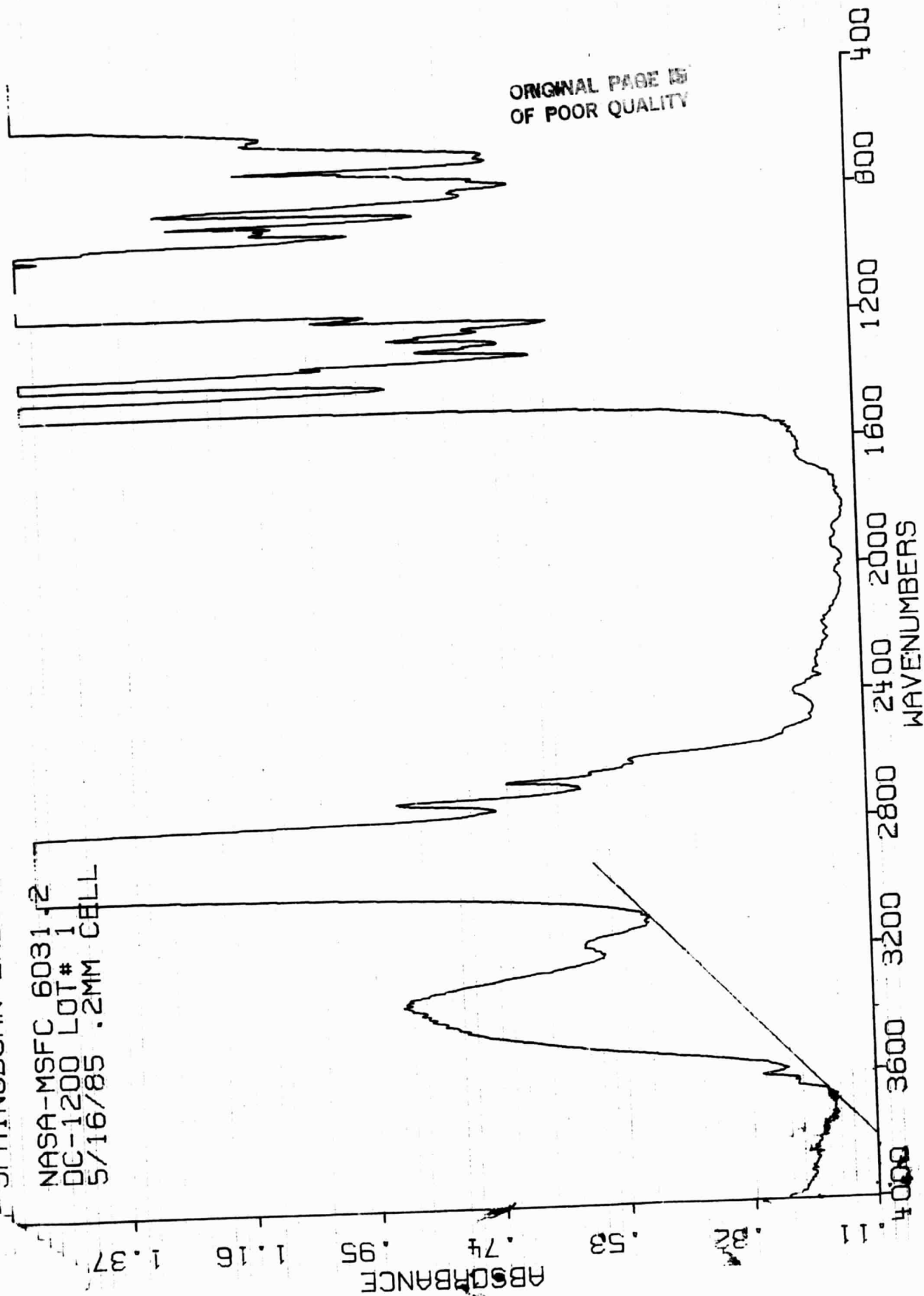


FIG. 20

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NASA-MSFC 6031-2
DC-1200 LOT# 1
5/16/85 .2MM CELL

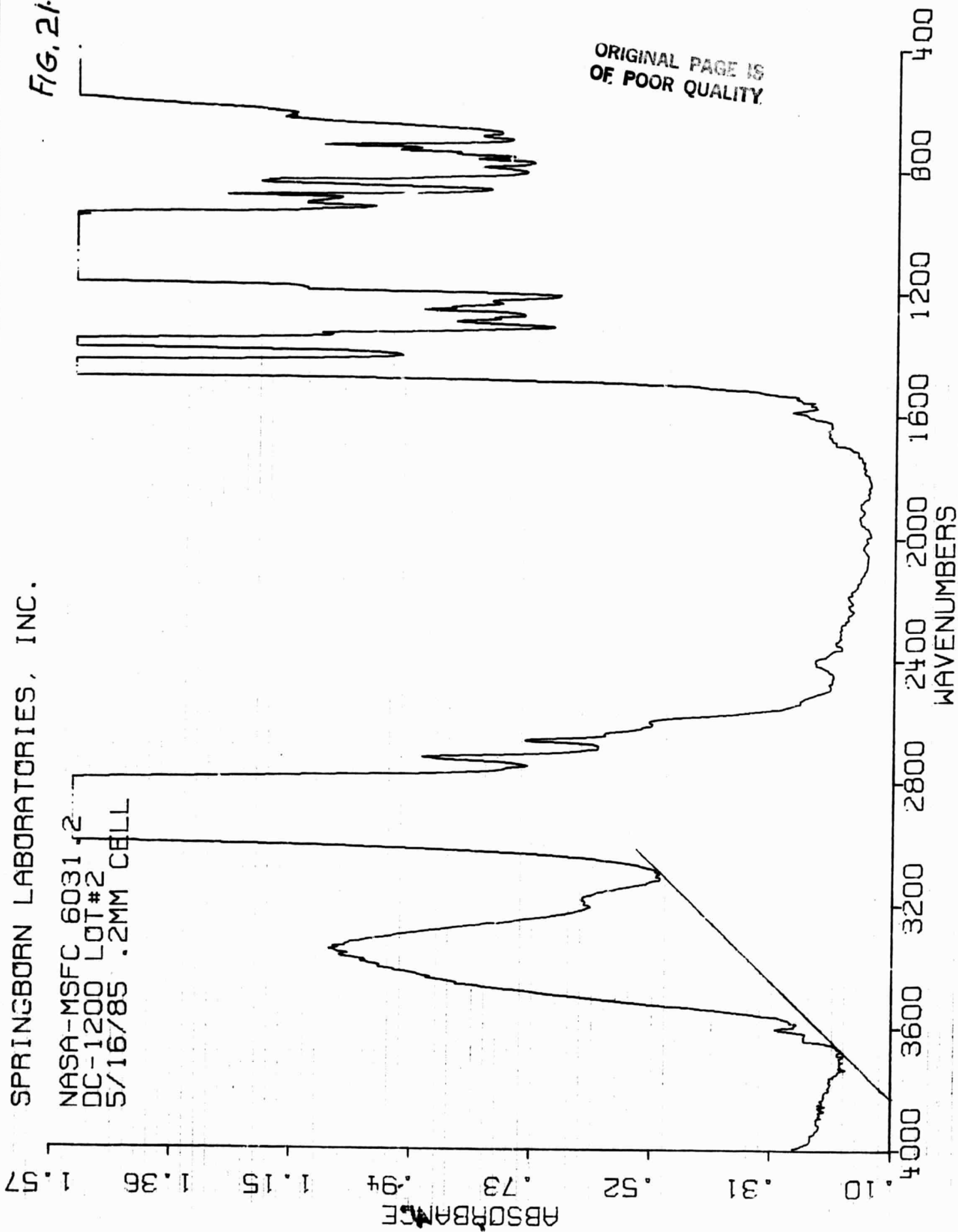


SPRINGBORN LABORATORIES, INC.

NASA-MSFC 6031.2
DC-1200 LOT#2
5/16/85 .2MM CELL

FIG. 21

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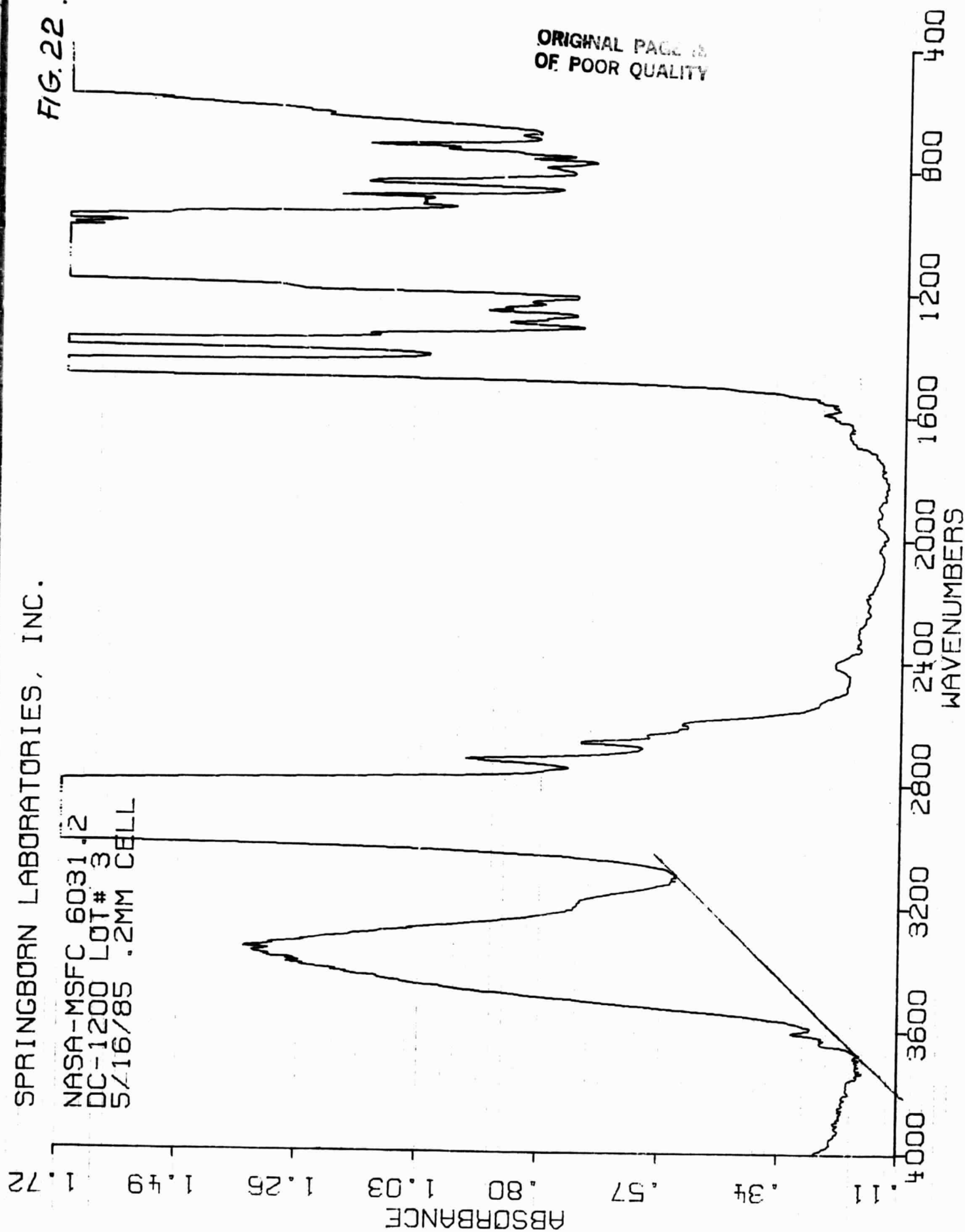


SPRINGBORN LABORATORIES, INC.

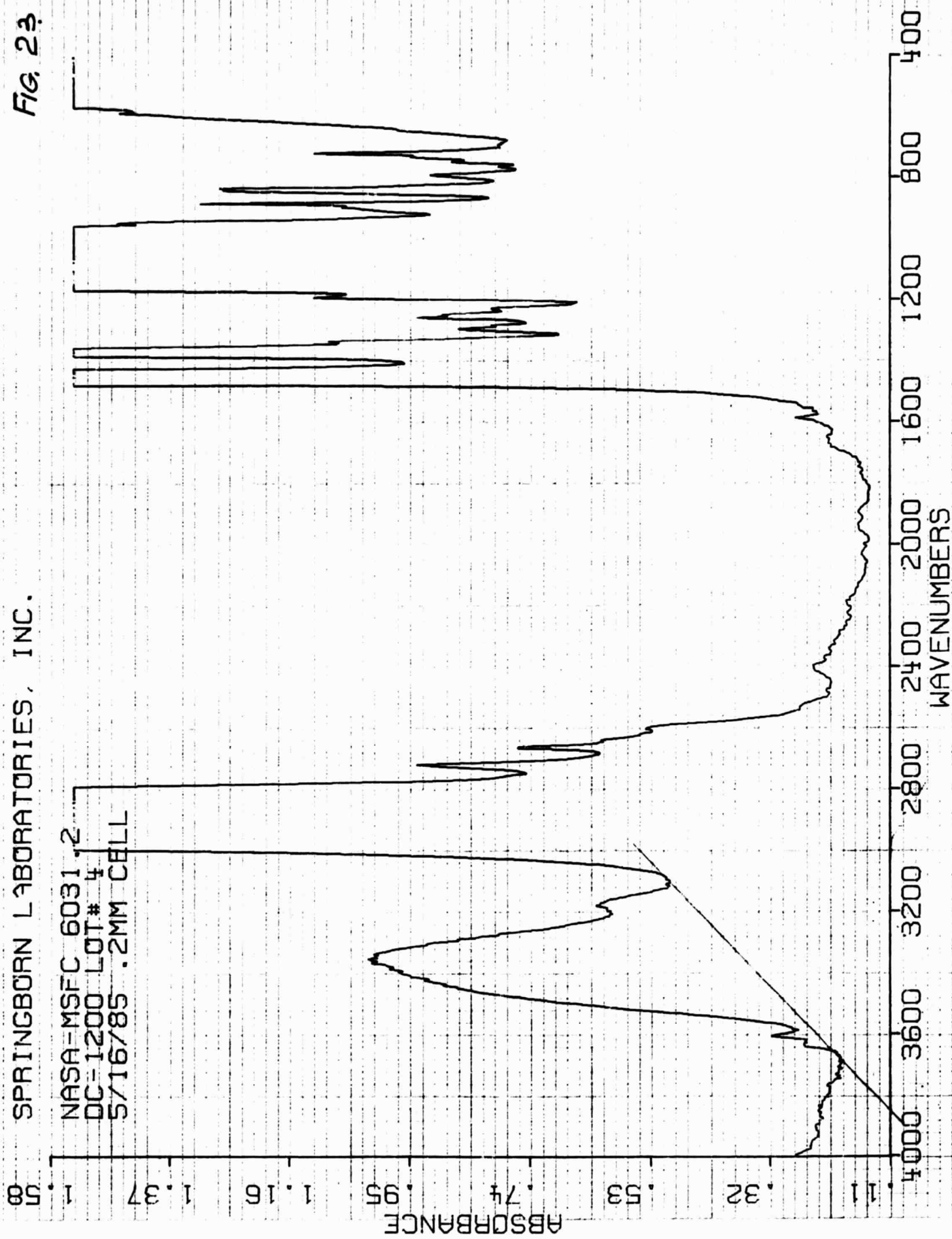
NASA-MSFC 6031.2
DC-1200 LOT# 3
5/16/85 .2MM CELL

FIG. 22.

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NASA-MSFC 6031.2
DC-1200 LOT# 4
5/16/85 .2MM CELL



SPRINGBORN LABORATORIES, INC.

NASA-MSFC 6031.2
DC-1200 LOT# 5
5/16/85 .2MM CELL

ABSORBANCE

1.5

1.3

1.1

.9

.7

.5

.3

.1

FIG. 24

WAVENUMBERS

4000

3600

3200

2800

2400

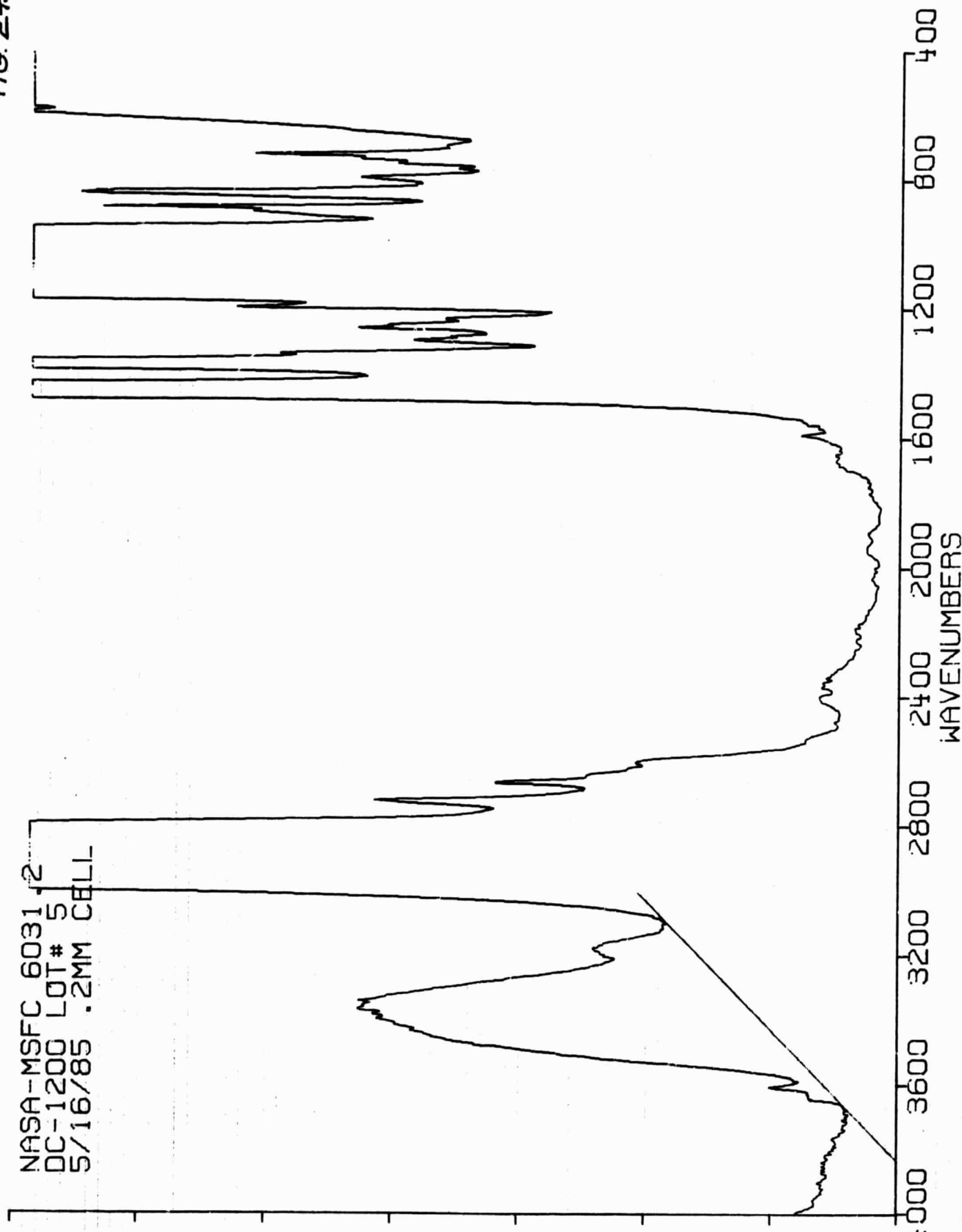
2000

1600

1200

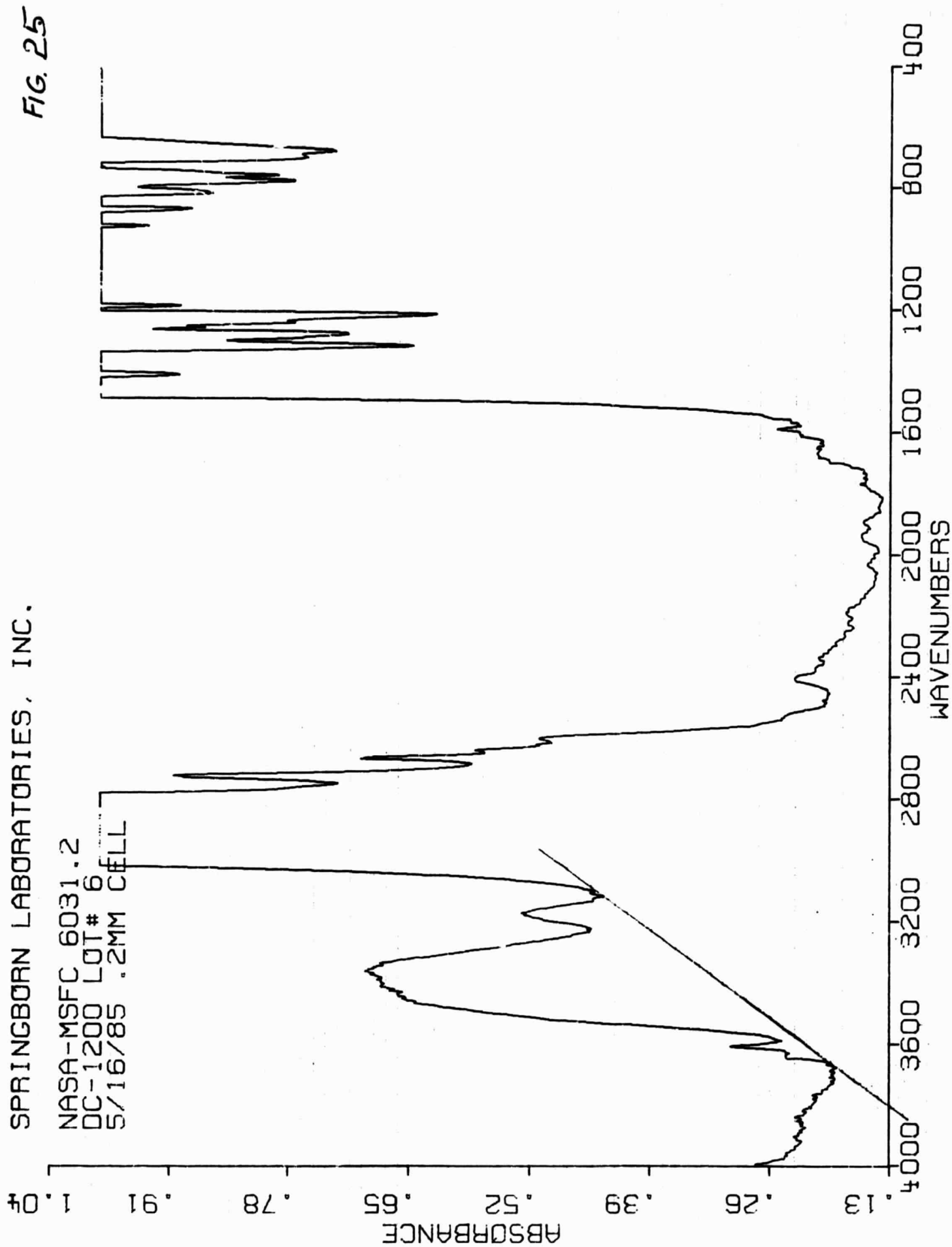
800

400



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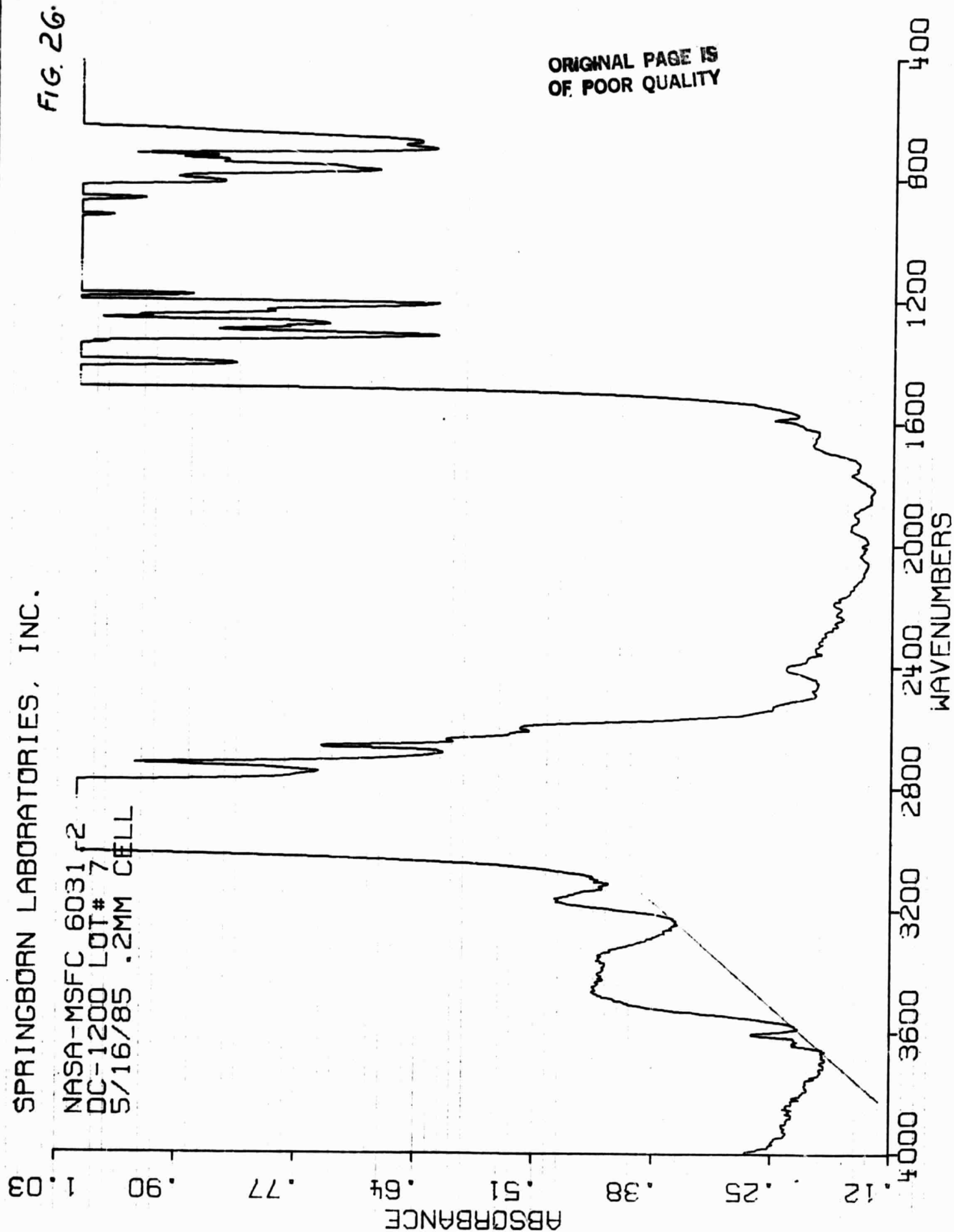
NASA-MSFC 6031.2
DC-1200 LOT# 6
5/16/85 .2MM CELL



SPRINGBORN LABORATORIES, INC.

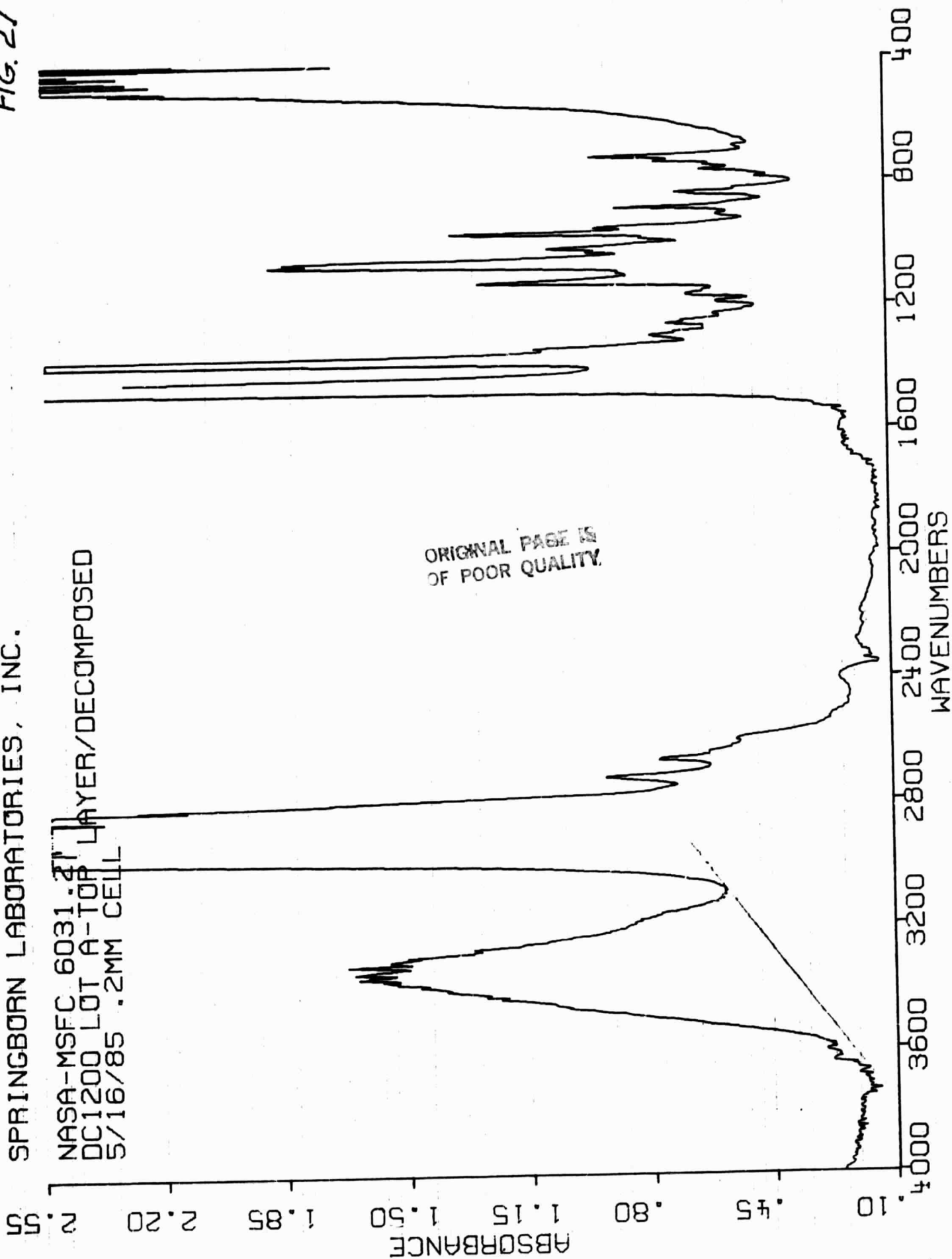
NASA-MSFC 6031 r2
DC-1200 LOT# 7
5/16/85 .2MM CELL

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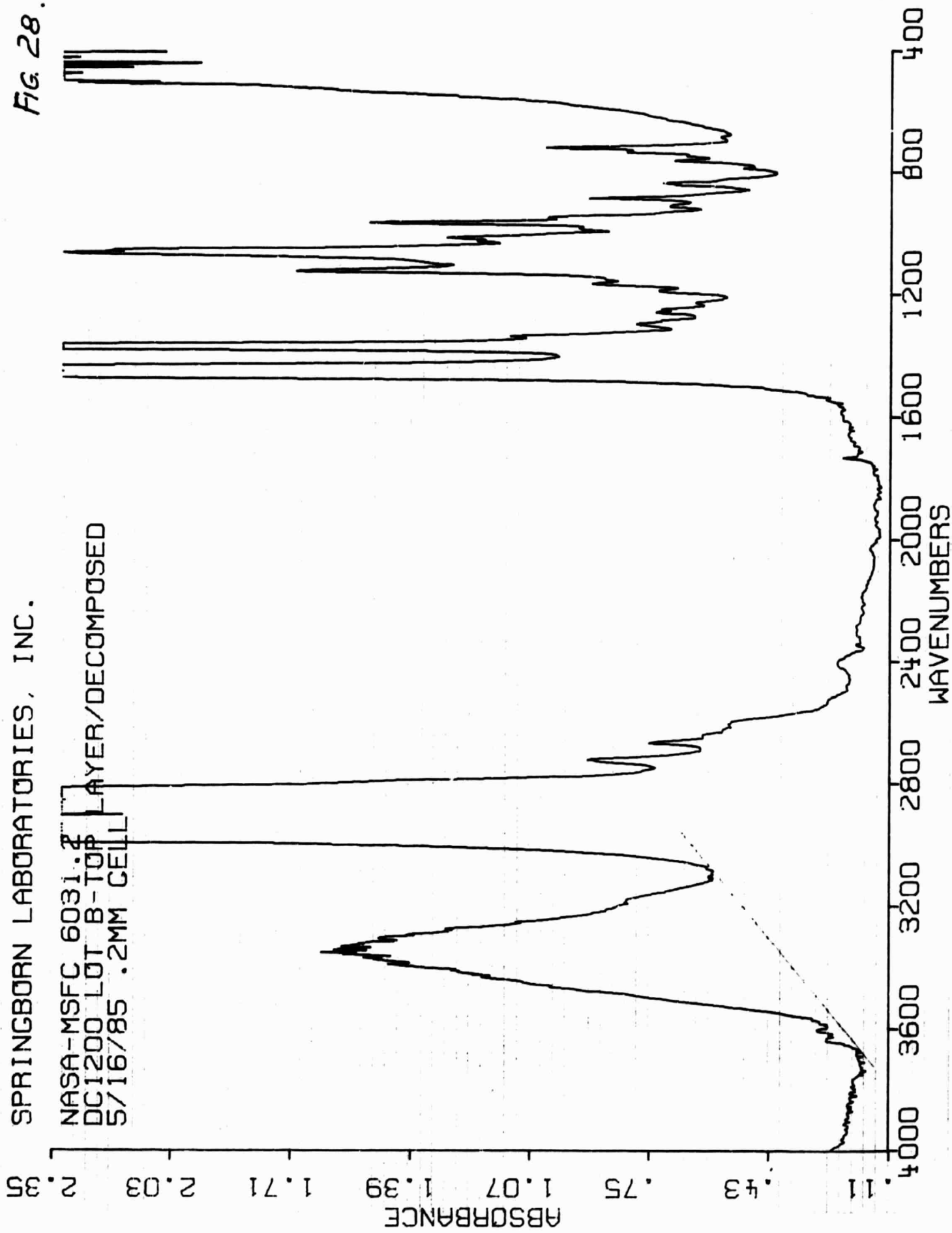
NASA-MSFC 6031-21
DC1200 LOT A-TOP LAYER/DECOMPOSED
5/16/85 .2MM CELL



SPRINGBORN LABORATORIES, INC.

FIG. 28.

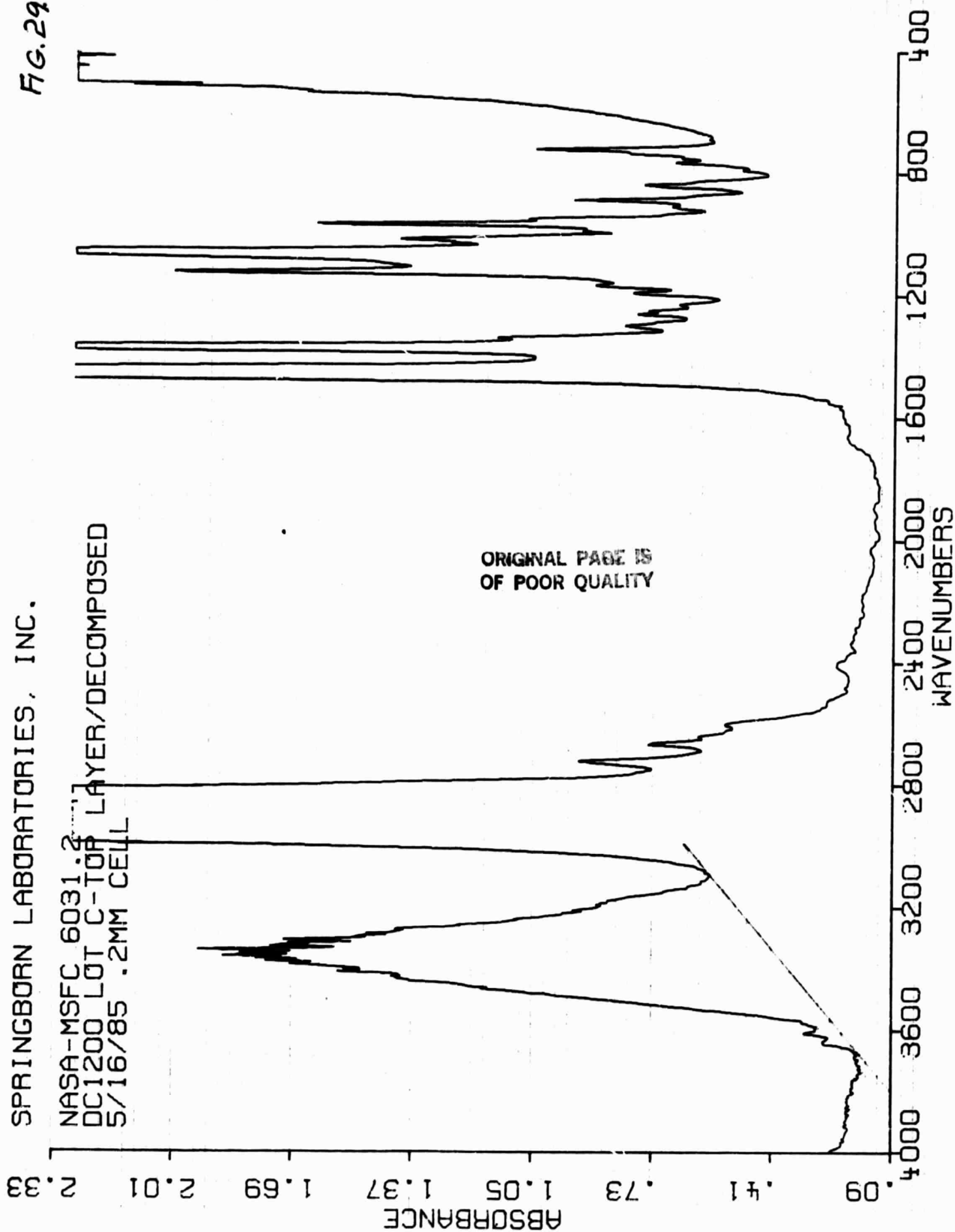
NASA-MSFC 6031.2
DC1200 LOT B-TOP LAYER/DECOMPOSED
5/16/85 .2MM CELL



SPRINGBORN LABORATORIES, INC.

FIG. 29

NASA-MSFC 6031.2
DC1200 LOT C-TOP LAYER/DECOMPOSED
5/16/85 .2MM CELL



IR SPECTRA VS SHEER TESTS

NASA-MSFC 6031.2

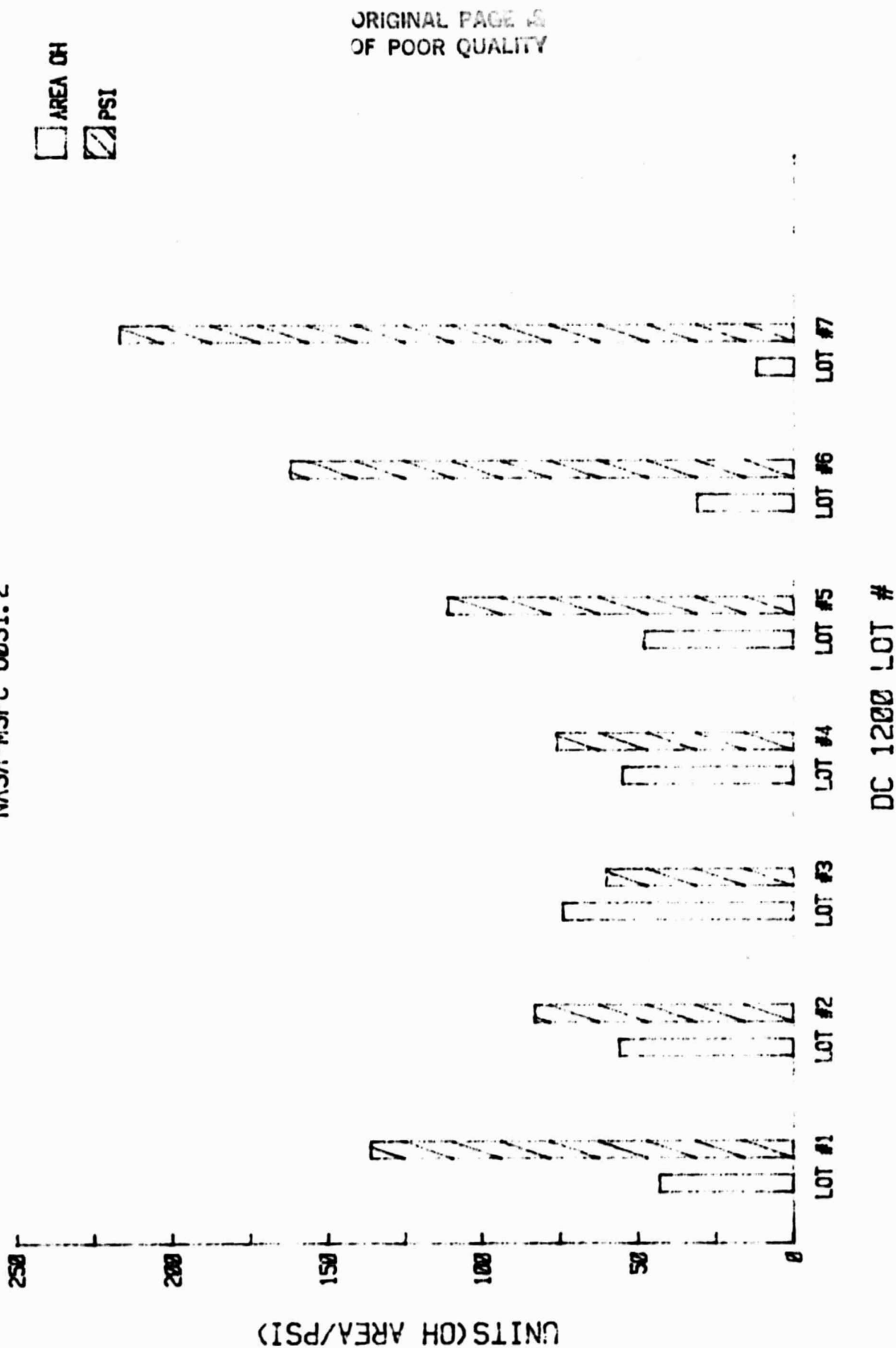


FIGURE 30

2007 RELEASE UNDER E.O. 14176

LAP SHEAR TESTS

COMMENTS:

NASA-MSFC
6031.2

01	2
02	2
03	2
04	2
05	1
06	1
07	1

NASA-MSFC 6031.2

LAP SHEAR TESTS

[illegible]

COMMENTS:

SERIES PANEL T

NASA-MSFC
6031.2

0	0	0	0	0	0	0
1	2	3	4	5	6	7
2	2	2	2	6	6	6

X

SERIES PANEL S

NASA - MSFC
6031.2

01	02	03	04	05	06	07
6	6	6	6	1	1	1

X

NASA-MSFC 6031.2

LAP SHEAR TESTS

[illegible]

COMMENTS:

NASA-MSFC
6031.2

56.346

o r	1
o v	1
o h	1
o s	1
o m	e
o t	e
o _	e

7

SAATCHI & SAATCHI LANCASHIRE & CO.

LAP SHEAR TESTS

COMMENTS:

COMMENTS:

NASA-MSFC
6031.2

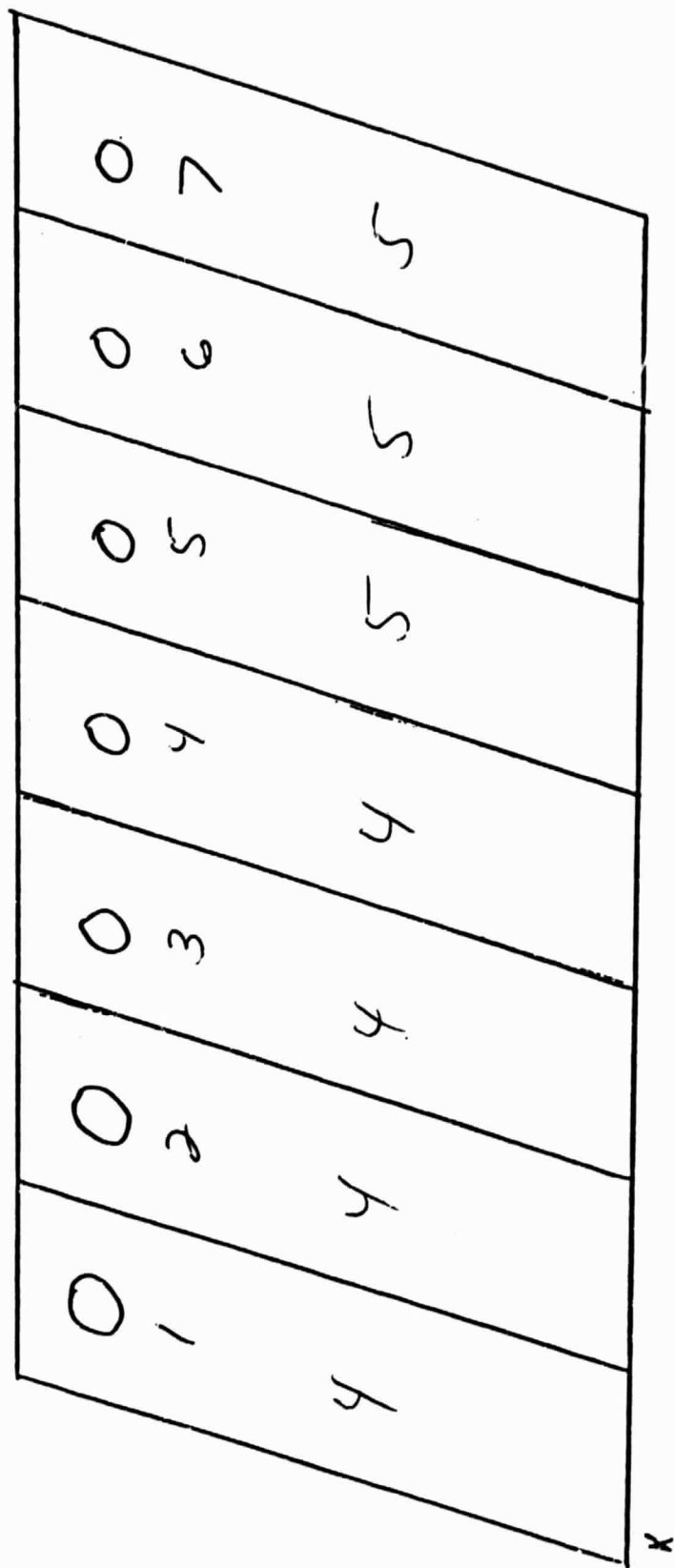
SERIES PANEL ✓

0	1	0	2	0	3	0	4	0	5	0	6	0	7
2	2	2	2	7	7	7	7	7	7	7	7	7	7

A

SERIES PANEL

3



NASA-MSCFC
6031.2

SERIES PANEL

y

0	0	0	0	0	0	0
7	6	5	4	3	2	1
5	5	5	5	4	4	4

x

NASA-MSFC
6031.2

SERIES PANEL X

0	0	0	0	0	0	0
1	2	3	4	5	6	7

SERIES PANEL Z

0 1	1
0 2	1
0 3	1
0 4	2
0 5	3
0 6	3
0 7	3
0 8	3

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NASA 6031.2
ASTM D1002 (MOD)
LAP SHEAR TEST
CROSS HEAD SPEED 0.05"/MIN
CHART SPEED 0.5"/MIN
5-30-85 HRP

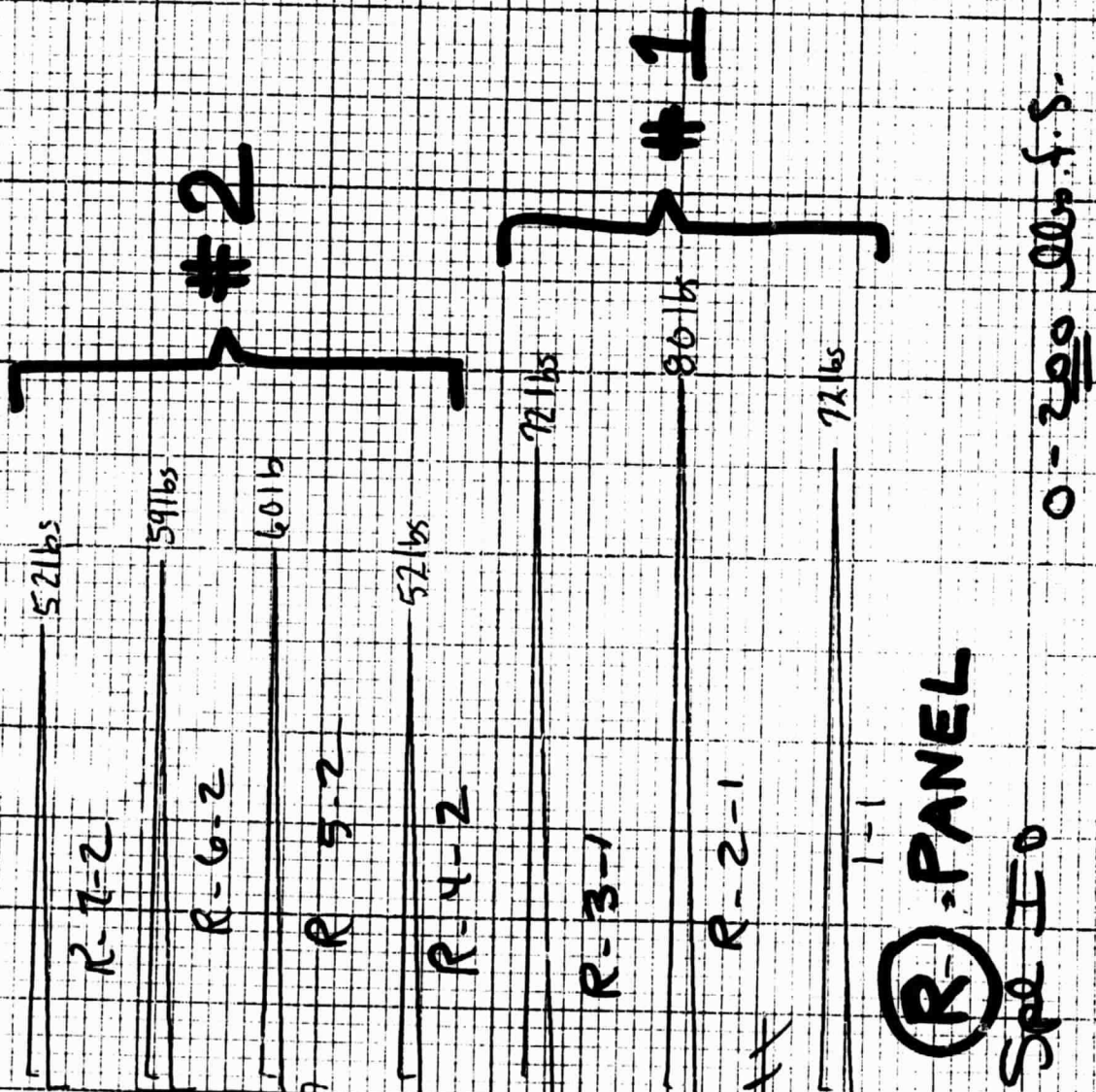
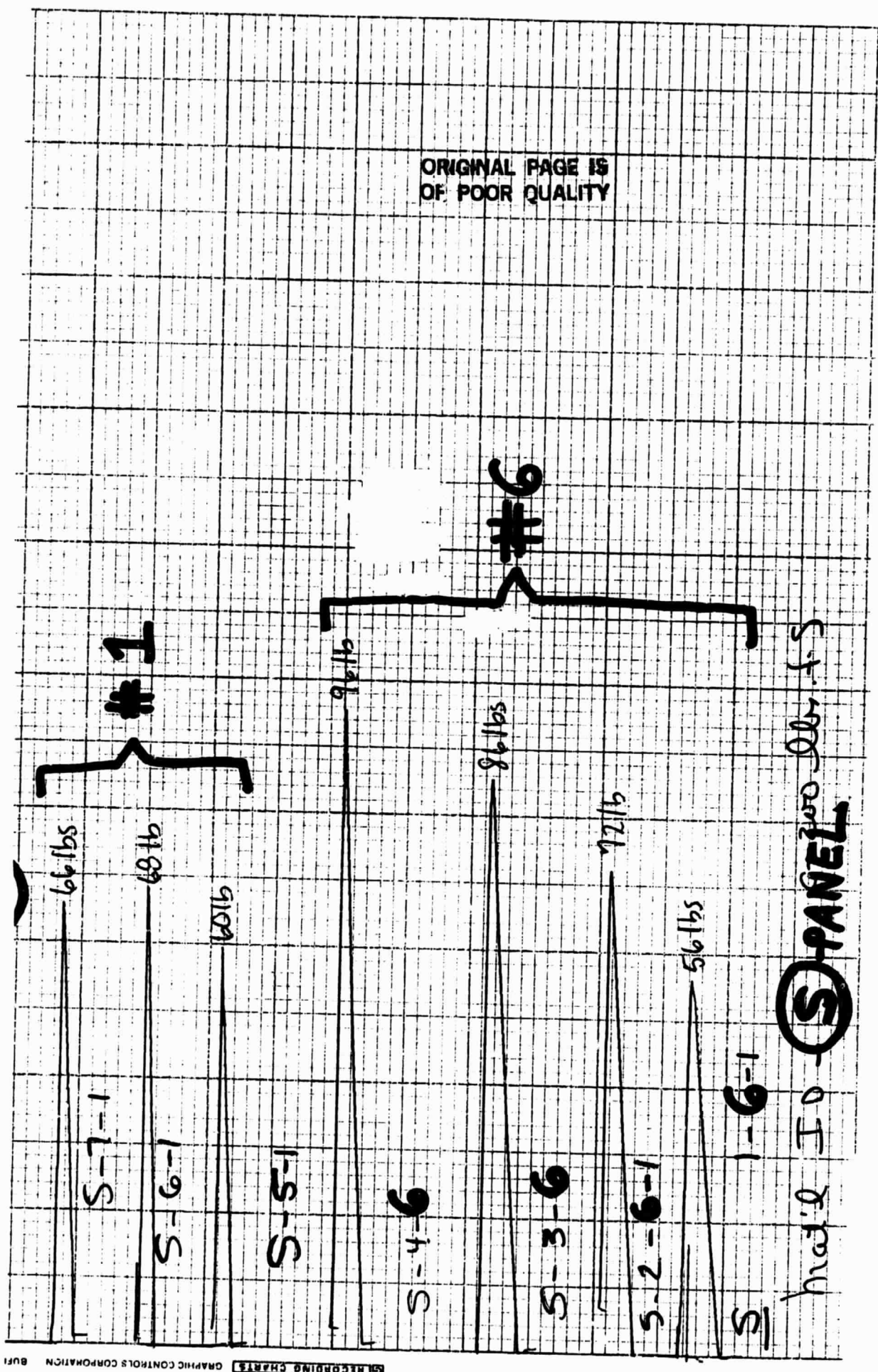
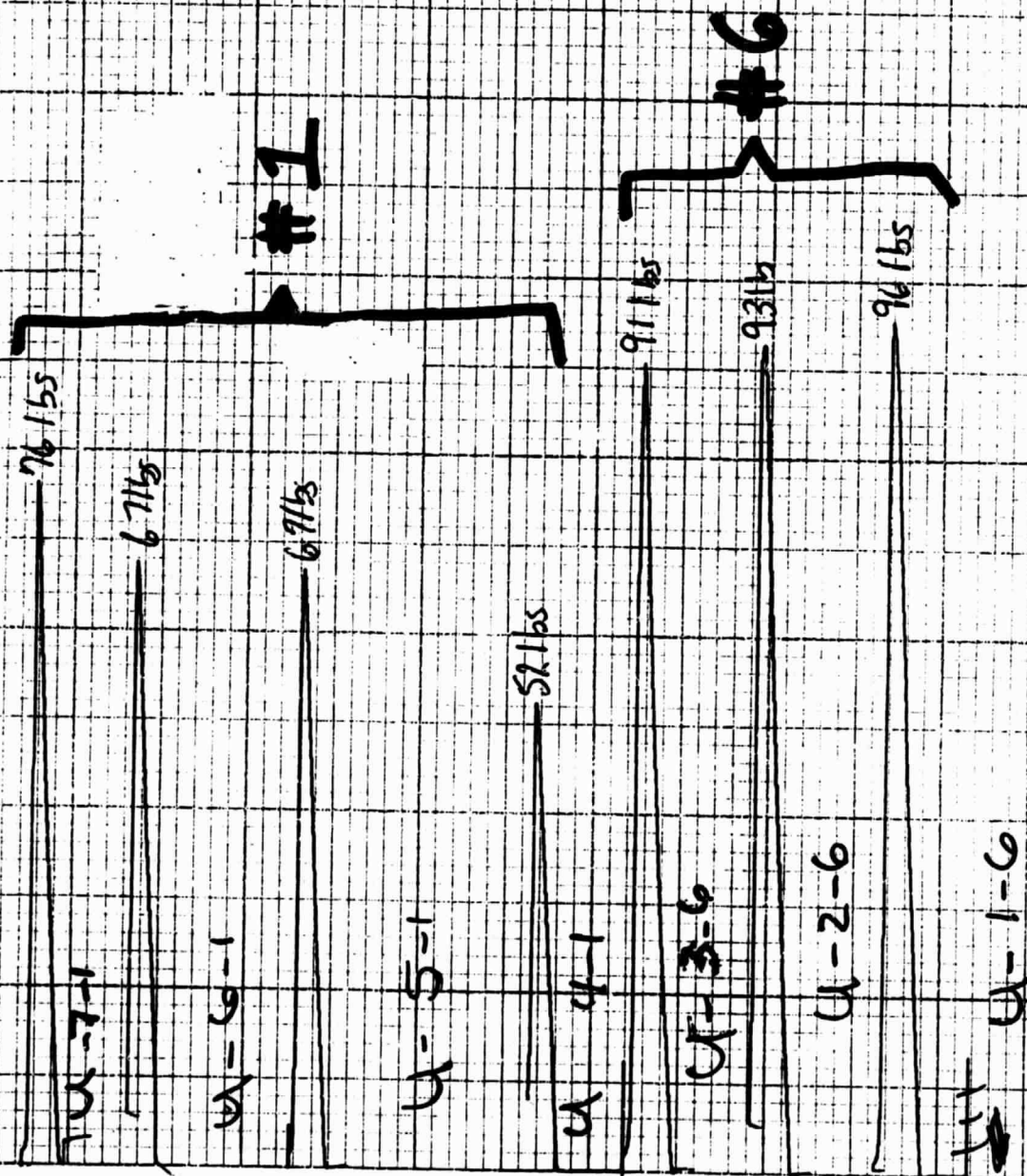


FIG. 41



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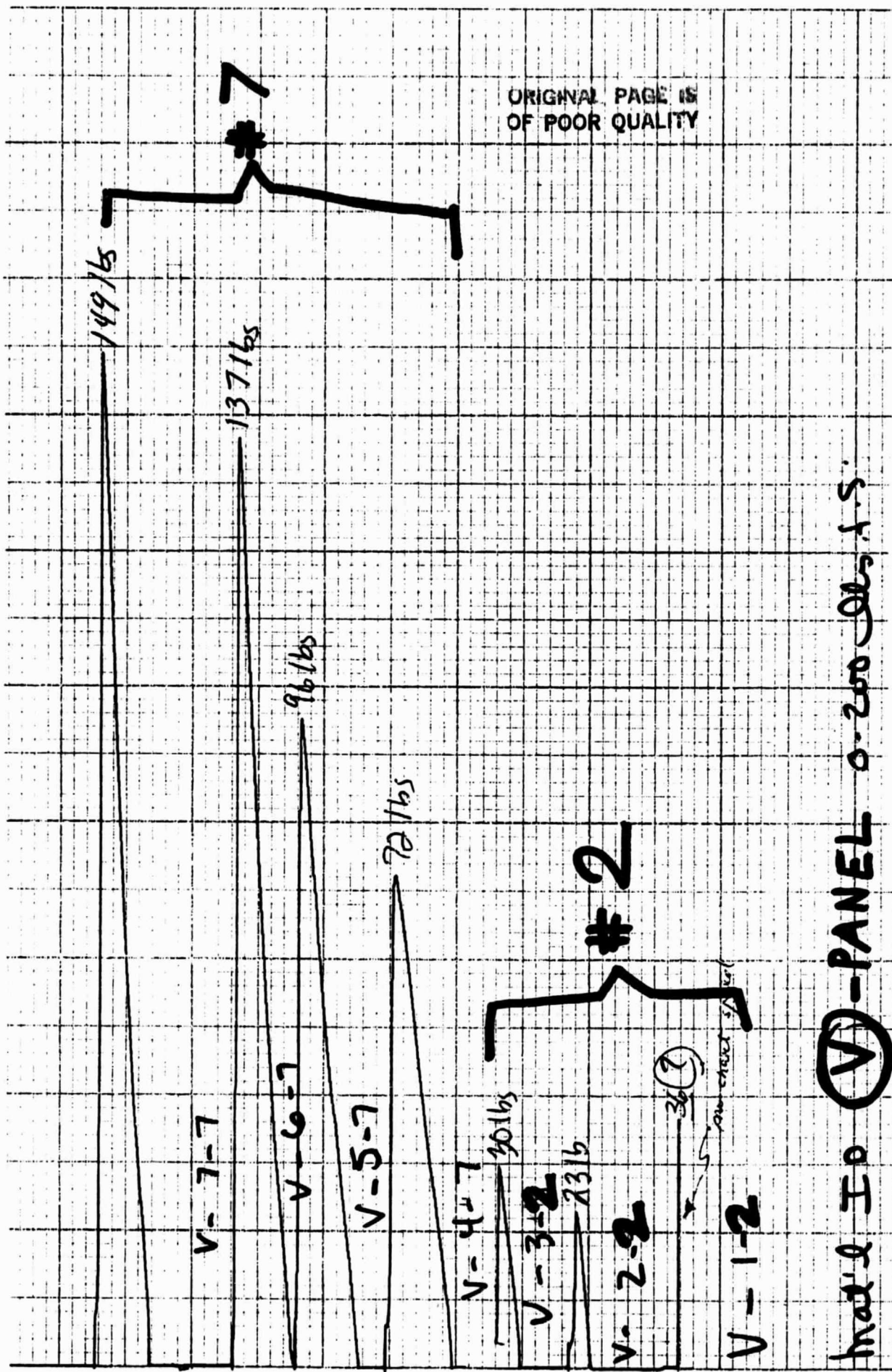
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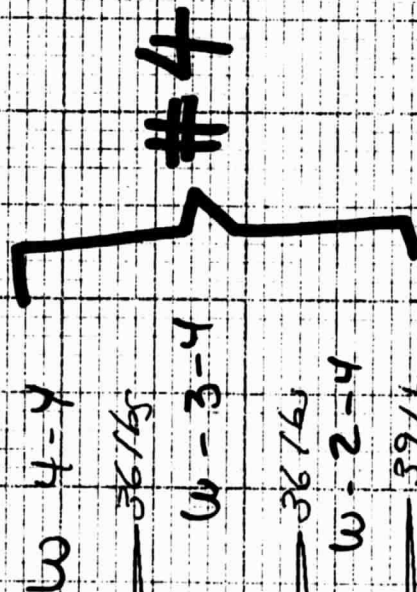
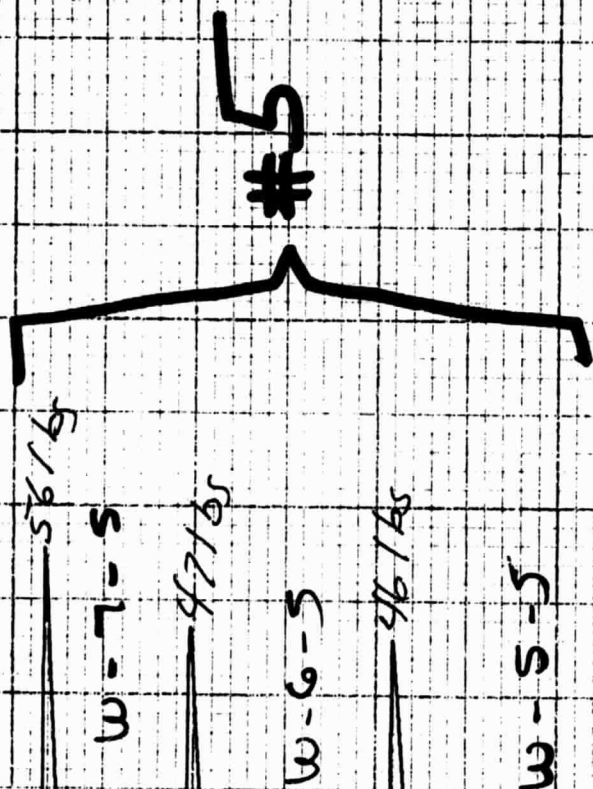
(0-200 lbs.)
fs

Spl ID

Mat 10 (U) - PANEL



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W-PANEL

Cross Head Speed 0.05"

Chart speed 0.5"

5-31-85 H.K.P.

Earl

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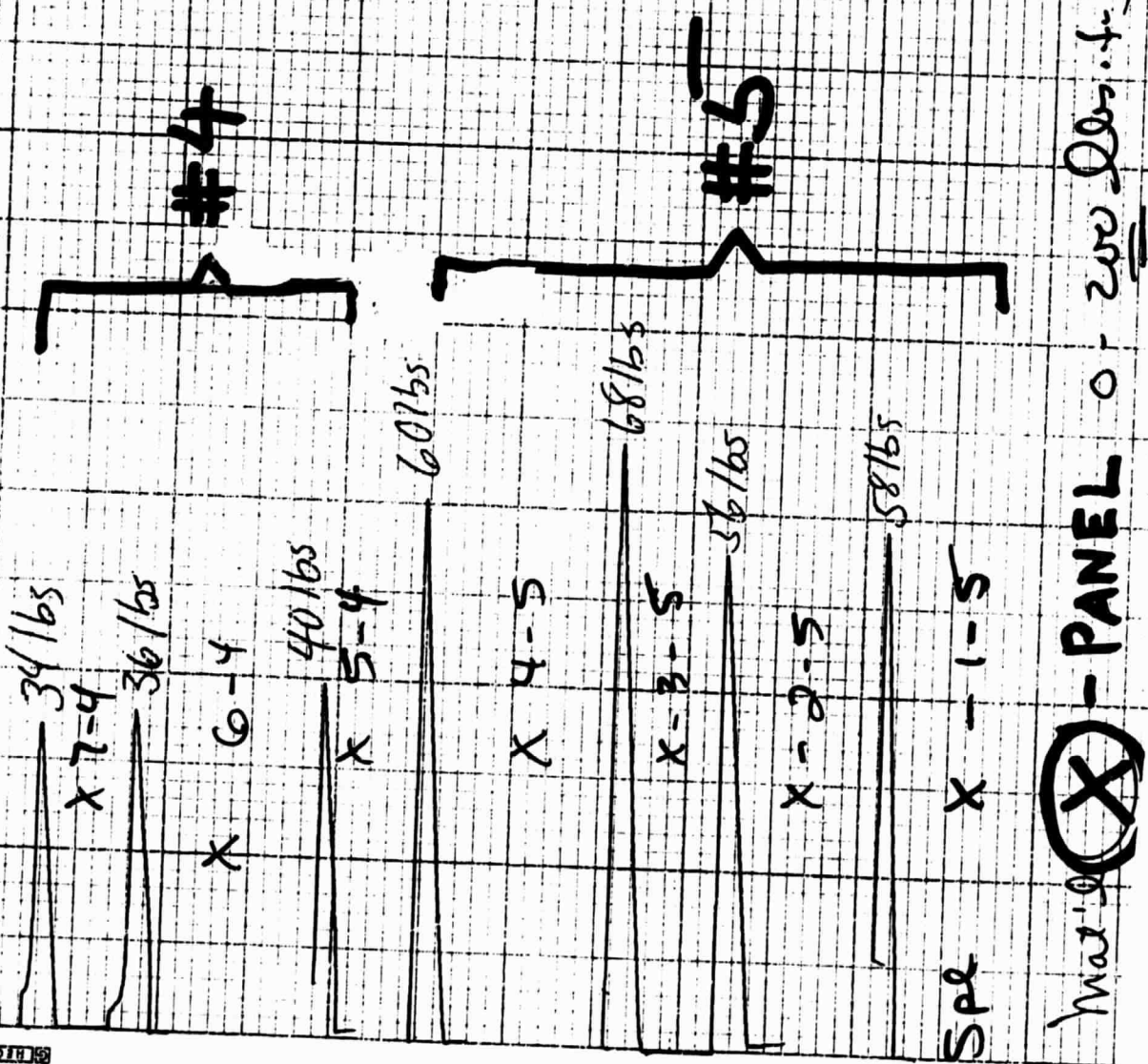
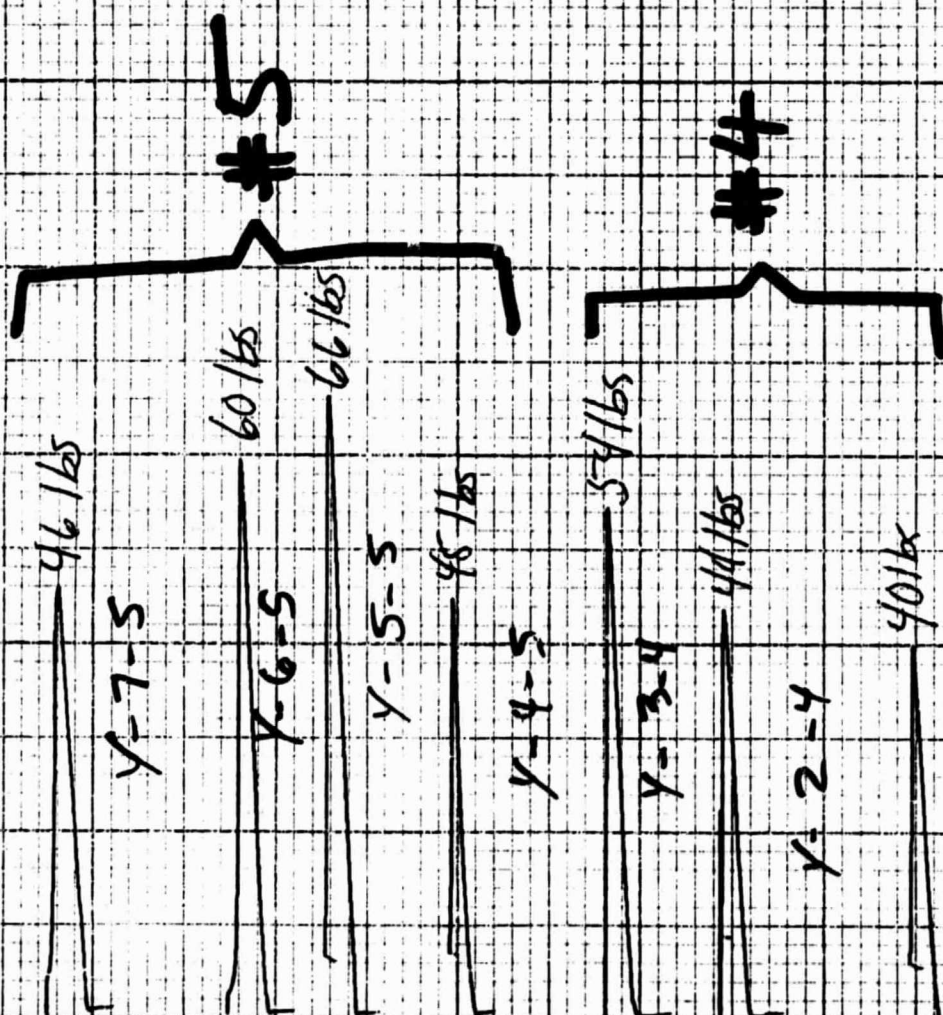


FIG. 47

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Y-PANEL

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